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☐ 1. Document ID: US 20010045360 A1 KR 2001107788 A CA 2349156 A1 EP 1164208 A2 JP 2002121699 A CN 1335419 A

L1: Entry 1 of 1 File: DWPI Nov 29, 2001

DERWENT-ACC-NO: 2002-066028
DERWENT-WEEK: 200236

TITLE: Electroplating of plating target articles involves utilizing combination of vibrational flow in plating bath and plating current of pulse

INVENTOR: OMASA, R

PRIORITY-DATA: 2001JP-0129994 (April 26, 2001), 2000JP-0155046 (May 25, 2000), 2000JP-0243249 (August 10, 2000)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
US 20010045360 A1	November 29, 2001		031	C25D005/18
KR 2001107788 A	December 7, 2001		000	C25D005/18
CA 2349156 A1	November 25, 2001	E	000	C25D005/18
EP 1164208 A2	December 19, 2001	E	000	C25D005/08
JP 2002121699 A	April 26, 2002		018	C25D021/10
CN 1335419 A	February 13, 2002		000	C25D005/18

INT-CL (IPC): C25 D 5/08; C25 D 5/18; C25 D 5/54; C25 D 7/00; C25 D 7/12; C25 D 17/00; C25 D 17/16; C25 D 21/10; C25 D 21/12; H01 L 21/288

Full | Title | CIT.1 | REV.1 | CLS.1 | REF.1 | SEQ.1 | ATT.1 | KWIC.1

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2001ep-0112689.ap.	1

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EAST Browser - L32: (1) 26 and 29 | EP 1164208 A2 | Tag: S | Doc: 1/1 (SORTED) | "Full" 1/33 (Total images 33)

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Document ID	Pages	1	2	3			U	S	C	P	Kind Codes	Source
EP 1164208 A2	33											EPO

PUB-NO: EP001164208A2

DOCUMENT-IDENTIFIER: EP 1164208 A2

TITLE: Electroplating method using combination of vibrational flow in plating bath and plating current of pulse

PUBN-DATE: December 19, 2001

INVENTOR-INFORMATION:

NAME	COUNTRY
OMASA, RYUSHIN	JP

ASSIGNEE-INFORMATION:

NAME	COUNTRY
JAPAN TECHNO CO LTD	JP

APPL-NO: EP01112689

APPL-DATE: May 25, 2001

PRIORITY-DATA: JP2000155046A (May 25, 2000) , JP2000243249A (August 10, 2000) , JP2001129994A (April 26, 2001)

INT-CL (IPC): C25D005/08, C25D005/18 , C25D021/10 , C25D017/00

ABSTRACT:

CHG DATE=20020103 STATUS=O> In an electroplating method, a plating target article (X) disposed so as to be in contact with plating bath (14) is set as a cathode while a metal member disposed so as to be in contact with the plating bath (14) is set as an anode, and a voltage is applied between the cathode and the anode while vibrational flow is induced by vibrating vibrational vanes (16f) which are fixed in multi-stage style to a vibrating rod (16e) vibrating in the plating bath (14) interlockingly with vibration generating means (16d). Plating current flowing from the anode through the plating bath (14) to the cathode is pulsed and alternately set to one of a first state where the plating current keeps a first value I1 for a first time T1 and a second state where the plating current keeps a second value I2 having the same polarity as the first value I1 for a second time T2, the first value I1 being five or more times larger than the second value I2, and the first time T1 being three or more times longer than the second time T2. <IMAGE>

(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 1 164 208 A2

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication: 19.12.2001 Bulletin 2001/51

(51) Int Cl.7: C25D 5/08, C25D 5/18, C25D 21/10, C25D 17/00

(21) Application number: 01112689.3

(22) Date of filing: 25.05.2001

(84) Designated Contracting States: AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE TR
Designated Extension States: AL LT LV MK RO SI

(30) Priority: 25.05.2000 JP 2000155046
10.08.2000 JP 2000243249
26.04.2001 JP 2001129994

(71) Applicant: Japan Techno Co., Ltd. Tokyo (JP)

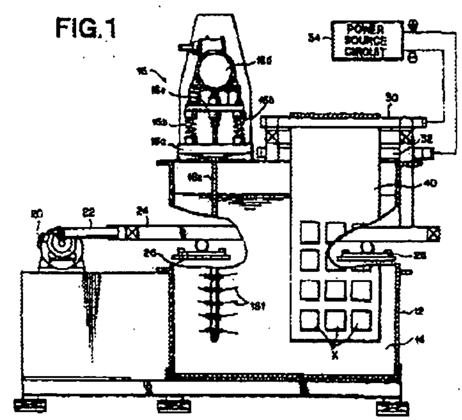
(72) Inventor: Omasa, Ryushin Fujisawa-shi, Kanagawa (JP)

(74) Representative: Gervasi, Gemma, Dr. et al
Notarbartolo & Gervasi S.p.A., Corso di Porta Vittoria, 9
20122 Milano (IT)

(54) **Electroplating method using combination of vibrational flow in plating bath and plating current of pulse**

(57) In an electroplating method, a plating target article (X) disposed so as to be in contact with plating bath (14) is set as a cathode while a metal member disposed so as to be in contact with the plating bath (14) is set as an anode, and a voltage is applied between the cathode and the anode while vibrational flow is induced by vibrating vibrational vanes (16f) which are fixed in multi-stage style to a vibrating rod (16e) vibrating in the plating bath (14) interlockingly with vibration generating means (16d). Plating current flowing from the anode through the plating bath (14) to the cathode is pulsed and alternately set to one of a first state where the plating current keeps a first value I1 for a first time T1 and a second state where the plating current keeps a second value I2 having the same polarity as the first value I1 for a second time T2, the first value I1 being five or more times larger than the second value I2, and the first time T1 being three or more times longer than the second time T2.

FIG.1



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  - L8: (17868) 12 or 15
  - L11: (201285) pulse or pulses or pulsed or pulsing
  - L14: (9477) pulsation or pulsations or pulsating
  - L17: (209643) 111 or 114
  - L20: (706535) current or voltage
  - L23: (24409) 117 near3 120
  - L26: (126) 18 and 123
  - L29: (22386) vane or vanes
  - L32: (1) 126 and 129
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  - L38: (131488) vibration or vibrations vibrational
  - L41: (148088) 135 or 138
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126 and 141

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	U	1	PT	P	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor	S	C	2	3		5
1	☑	☐	☐	☐	JP 63312996 A	19881221	2	METHOD FOR ELECTRODEPOSITING AMORPHOUS ALLOY				WATANABE, SHUNJI et al.	☐	☐	☐	☐	☐	☐
2	☑	☐	☐	☐	EP 1164208 A2	20011219	33	Electroplating method using combination of vibrational				OMASA, RYUSHIN	☐	☐	☐	☐	☐	☐

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  - L3: (22060) pulsation or pulsating
  - L4: (991368) current or voltage
  - L5: (388475) 12 or 13
  - L6: (82453) 15 near3 14
  - L7: (15) 11 and 16
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11 and 16

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	U	1	PT	P	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor	S	C	2	3		
1	☑	☐	☐	☐	US 6251250 B1	20010626	17	Method of and apparatus for controlling fluid flow and	205/89	204/224R; 204/230.2;		Keigler, Arthur	☐	☐	☐	☐	☐	☐
2	☑	☐	☐	☐	US 6235181 B1	20010522		Method of operating process for anodizing valve metals	205/148	205/234; 205/322;		Kinard, John T. et al.	☐	☐	☐	☐	☐	☐
3	☑	☐	☐	☐	US 6132587 A	20001017		Uniform electroplating of wafers	205/123	204/224R; 204/229.6;		Jorne, Jacob et al.	☐	☐	☐	☐	☐	☐
4	☑	☐	☐	☐	US 5958604 A	19990928		Electrolytic process for cleaning and coating	428/612	205/102; 205/131;		Riabkov, Vitalig M. et al.	☐	☐	☐	☐	☐	☐
5	☑	☐	☐	☐	US 5911863 A	19990615		Method of manufacturing plastic foils which are	205/103	205/104; 205/118;		Vetter, Johann et al.	☐	☐	☐	☐	☐	☐
6	☑	☐	☐	☐	US 5700366 A	19971223		Electrolytic process for cleaning and coating	205/87	205/102; 205/131;		Steblianko, Valerij Leontievich et al.	☐	☐	☐	☐	☐	☐
7	☑	☐	☐	☐	US 5584983 A	19961217		Method for the production of a metal foam	205/103	205/104; 205/148;		Pruyn, Wilhelmus A.	☐	☐	☐	☐	☐	☐
8	☑	☐	☐	☐	US 5518605 A	19960521		Method of nitriding ferrous metal parts having improved	205/148	205/231; 205/320		Hadj-Rabah, Hocine et al.	☐	☐	☐	☐	☐	☐
9	☑	☐	☐	☐	US 4898647 A	19900206		Process and apparatus for electroplating copper foil	205/108	204/216; 204/229.4;		Luce, Betty M. et al.	☐	☐	☐	☐	☐	☐
10	☑	☐	☐	☐	US 4855020 A	19890808		Apparatus and method for the electrolytic plating of	205/137	204/212; 204/218;		Sirbola, Michael A.	☐	☐	☐	☐	☐	☐
11	☑	☐	☐	☐	US 4652348 A	19870324		Method for the production of alloys possessing high	205/104	204/DIG.9; 205/148;		Yahalom, Joseph et al.	☐	☐	☐	☐	☐	☐
12	☑	☐	☐	☐	US 4496436 A	19850129		Pulse electrodepositing method	205/83	204/DIG.9; 205/103;		Inoue, Kiyoshi	☐	☐	☐	☐	☐	☐

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EAST Browser - L7: (15) 1 and 6 | US 6132587 A | Tag: S | Doc: 3/15 (SORTED) | Format : KWIC

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	Document ID	Pages	1	2	3	4	5	U	S	C	P	Kind Codes	Sou
1	US 6251250 B1	17							☑				USPAT
2	US 6235181 B1	6							☑				USPAT
3	US 6132587 A	12							☑				USPAT
4	US 5958604 A	17						☑					USPAT
5	US 5911863 A	7						☑					USPAT
6	US 5700366 A	12						☑					USPAT
7	US 5584983 A	6						☑					USPAT

US-PAT-NO: 6132587

DOCUMENT-IDENTIFIER: US 6132587 A

TITLE: Uniform electroplating of wafers

---------- KWIC ---------

Claims Text - CLTX (35):

means for applying pulsed current to said pump during the electroplating process.

Claims Text - CLTX (87):

37. A method according to claim 21 wherein said step of passing a current comprises pulsing said current .

Current US Cross Reference Classification - CCXR (6):

205/148

United States Patent [19]

Jorné et al.

[11] Patent Number: 6,132,587

[45] Date of Patent: Oct. 17, 2000

[54] UNIFORM ELECTROPLATING OF WAFERS

[75] Inventors: Jacob Jorné; Judith Ann Love, both of 359 Westminster Rd., Rochester, N.Y. 14607

[21] Appl. No.: 09/174,337

[22] Filed: Oct. 19, 1998

[51] Int. Cl.^{7} C25D 5/02; C25D 5/08; C25D 5/20; C25D 17/00; C25D 15/00

[52] U.S. Cl. .... 205/123; 205/133; 205/148; 205/157; 204/224 R; 204/263; 204/229.6; 204/DIG. 7

[58] Field of Search .... 204/224 R, 212, 204/229.6, DIG. 7, 263; 205/123, 137, 133, 103, 148, 157

[56] References Cited

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4,304,641	12/1981	Grandia et al.	204/DIG. 7
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5,445,172	8/1995	Thompson et al.	134/153
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M. Witty, S.P. Murarka and D.B. Fraser, SRC Workshop on Copper Interconnect Technology, Semiconductor Research Corporation, Research Triangle Park, NC, Aug. 17–18, 1993.

VLSI Multilevel Interconnection Conference, VMCI, Santa Clara, CA, Jun. 10–12, 1997.

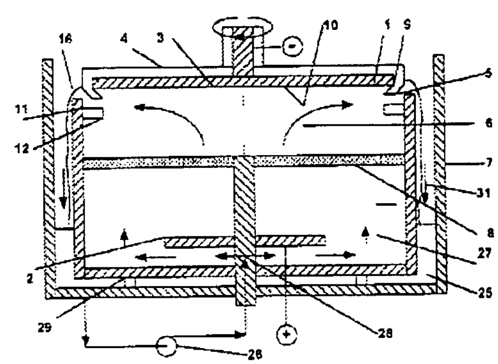
Primary Examiner—Donald R. Valentine

Attorney, Agent, or Firm—Fiehr Hohbach Test Albritton & Herbert LLP

[57] ABSTRACT

The non-uniformity of electroplating on wafers is due to the appreciable resistance of the thin seed layer and edge effects. Mathematical analysis of the current distribution during wafer electroplating reveals that the ratio between the resistance of the thin deposited seed layer and the resistance of the electrolyte and the electrochemical reaction determines the uniformity of the electroplated layer. Uniform plating is critical-in-wafer metallization for the subsequent step of chemical mechanical polishing of the wafer. Based on the analysis, methods to improve the uniformity of metal electroplating over the entire wafer include increasing the resistance of the electrolyte, increasing the distance between the wafer and the anode, increasing the thickness of the seed layer, increasing the ionic resistance of a porous separator placed between the wafer and the anode, placement of a rotating distributor in front of the wafer, and establishing contacts at the center of the wafer. The rotating distributor generates multiple jets hitting the surface of the wafer, thus ensuring conformal electroplating. The jets can be either submerged in the electrolyte or above the level of the electrolyte. The shape and uniformity of the electroplated layer can be also determined by the shape and relative size of the counter-electrode (anode), by masking the edge of the wafer and by periodically reversing the plating current. The problem of uniformity is more severe as the diameter of the wafer becomes larger.

37 Claims, 5 Drawing Sheets



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	Document ID	Pages	1	2	3		5	U	S	C	P	Kind Codes	Sou
7	US 5584983 A	6							x				USPAT
8	US 5518605 A	6							x				USPAT
9	US 4898647 A	11							x				USPAT
10	US 4855020 A	24							x				USPAT
11	US 4652348 A	6							x				USPAT
12	US 4496436 A	9							x				USPAT
13	US 4466864 A	8						x					USPAT

US-PAT-NO: 4496436

DOCUMENT-IDENTIFIER: US 4496436 A

TITLE: Pulse electrodepositing method

---------- KWIC ---------

Abstract Text - ABTX (1):

Electrodeposition is carried out using an electric current which is applied in the form of pulses between a workpiece and an electrode in an electrolyte from which metal is to be deposited upon the workpiece. The pulses have a pulse duration not greater than 100 microseconds and the pulse duration is preferably between 1-50 microseconds while the off time or interval between adjacent pulses is more than twice as long as the on time of the pulses.

Brief Summary Text - BSTX (2):

There has become known in the art a pulse depositing method in which an electric current in the form of a series of pulses is passed between a substrate or workpiece and an electrode spacedly juxtaposed therewith in the presence of an electrolyte. Such pulse depositing techniques are described, for example, in Japanese Patent Specifications No. 40-8801 published May 8, 1965 and No. 48-5529 published Feb. 17, 1973. As noted therein, pulse depositing is advantageous in that it enables an efficient ion-control in the depositing electrolyte which permits the deposition process to be achieved at an increased rate and precision. These effects are further enhanced when the liquid electrolyte is supplied so as to pass through the depositing zone at an elevated flow rate, thereby permitting the deposition to be carried out at an increased current density.

Brief Summary Text - BSTX (5):

In accordance with the present invention, there is provided a method of electrodeposition in which an electric current in the form of pulses is applied between a workpiece and an electrode juxtaposed therewith in the presence of a liquid electrolyte to form an electrolytically deposited layer on the workpiece, the pulses having a pulse duration not greater than 100 microseconds.

Brief Summary Text - BSTX (11):

The electrical signal representative of the gap condition may be used to modify the basic switching control pulses in the manner to alter a parameter of the electrodepositing current pulses, i.e. pulse on time, off time and/or amplitude, in accordance with the gap condition so that the electrodepositing operation may proceed under an optimum condition.

Detailed Description Text - DETX (10):

The switch 12 is turned on and off by a pulser 16 which provides control

United States Patent [19]

Inoue

[11] Patent Number: 4,496,436

[45] Date of Patent: Jan. 29, 1985

[54] PULSE ELECTRODEPOSITING METHOD

[75] Inventor: Kiyoshi Inoue, Tokyo, Japan

[73] Assignee: Inoue-Japax Research Incorporated, Yokohama, Japan

[21] Appl. No.: 973,608

[22] Filed: Dec. 27, 1978

Related U.S. Application Data

[63] Continuation of Ser. No. 828,502, Aug. 29, 1977, abandoned.

[30] Foreign Application Priority Data

Sep. 1, 1976 [JP] Japan 51-104724
Dec. 14, 1976 [JP] Japan 51-150051
Jun. 10, 1977 [JP] Japan 52-69129

[51] Int. Cl.³ C25D 5/18

[52] U.S. Cl. .... 204/23; 204/45.1; 204/49; 204/52 R; 204/DIG. 9

[58] Field of Search .... 204/DIG. 9, 49, 52 R, 204/45 R, 23

[56] References Cited

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3,959,088 5/1976 Sullivan .... 204/14 R
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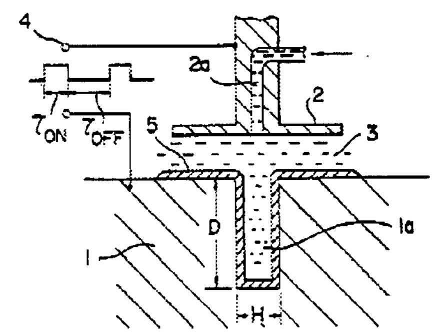
W. Sullivan, Plating, vol. 62, No. 2, pp. 139-141, (1975).

Primary Examiner—G. L. Kaplan
Attorney, Agent, or Firm—Karl F. Ross; Herbert Dubno

[57] ABSTRACT

Electrodeposition is carried out using an electric current which is applied in the form of pulses between a workpiece and an electrode in an electrolyte from which metal is to be deposited upon the workpiece. The pulses have a pulse duration not greater than 100 microseconds and the pulse duration is preferably between 1-50 microseconds while the off time or interval between adjacent pulses is more than twice as long as the on time of the pulses.

8 Claims, 9 Drawing Figures



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  - L3: (22060) pulsation or pulsating
  - L4: (991368) current or voltage
  - L5: (388475) l2 or l3
  - L6: (82453) l5 near3 l4
  - L7: (15) l1 and l6
  - L8: (424) (204/273).CCLS.
  - L9: (31937) electroplat$ or electrodeposit$
  - L10: (5361) (electrolytic$ or electrochemical$) near1 deposit$
  - L11: (34850) l9 or l10
  - L12: (129) l8 and l11
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1					US 6368482 B1	20020409	27	Plating processes utilizing high intensity acoustic	205/91	204/222; 204/273;		Oeftering, Richard C. et al.		
2					US 6277265 B1	20010821		Apparatus and method for electrocoriolysis, the	205/687	204/242; 204/267;		Hanak, Joseph J.		
3					US 6261433 B1	20010717		Electro-chemical deposition system and method of	205/96	204/230.2; 204/230.7;		Landau, Uziel		
4					US 6251250 B1	20010626	17	Method of and apparatus for controlling fluid flow and	205/89	204/224R; 204/230.2;		Keigler, Arthur		
5					US 5858199 A	19990112		Apparatus and method for electrocoriolysis the	205/687	204/242; 204/267;		Hanak, Joseph J.		
6					US 5173169 A	19921222		Electroplating method and apparatus	205/91	204/222; 204/273;		Garrison, Alexander J. et al.		
7					US 4584081 A	19860422		Apparatus for depositing metal on the rubbing parts	204/272	204/273; 204/277		Coulon, Andre		
8					US 4368106 A	19830111		Implantation of electrical feed-through conductors	205/123	204/273; 205/125;		Anthony, Thomas R.		
9					US 4329209 A	19820511		Process using an oxidant depolarized solid polymer	205/512	204/222; 204/273;		Johnson, Harlan B.		
10					US 4316786 A	19820223		Apparatus for electroplating particles of small dimension	204/223	204/229.4; 204/273;		Yu, Conrad M. et al.		
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12					US 3652442 A	19720328		ELECTROPLATING CELL INCLUDING MEANS TO AGITATE	204/273	204/261		Powers, John V. et al.		

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  - L14: (9477) pulsation or pulsations or pulsating
  - L17: (209643) 111 or 114
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  - L26: (126) 18 and 123
  - L29: (22386) vane or vanes
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1	☑	☐	☐	☐	EP 1164208 A2	20011219	33	Electroplating method using combination of vibrational				OMASA, RYUSHIN	☐	☐	☐	☐	☐	☐

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1	☑	☐	☐	☐	US 6261435 B1	20010717	18	Plating method	205/205	205/137; 205/148;		Omasa, Ryushin
2	☑	☐	☐	☐	US 6251250 B1	20010626		Method of and apparatus for controlling fluid flow and	205/89	204/224R; 204/230.2;		Keigler, Arthur
3	☑	☐	☐	☐	US 6221437 B1	20010424		Heated workpiece holder for wet plating bath	427/430.1	205/148; 205/209;		Reynolds, H. Vincent
4	☑	☐	☐	☐	US 6077412 A	20000620		Rotating anode for a wafer processing chamber	205/143	204/212; 204/224M;		Ting, Chiu H. et al.
5	☑	☐	☐	☐	US 5904827 A	19990518		Plating cell with rotary wiper and megasonic	205/68	204/263; 205/148		Reynolds, H. Vincent
6	☑	☐	☐	☐	US 5683564 A	19971104		Plating cell and plating method with fluid wiper	205/68	204/212; 204/238;		Reynolds, H. Vincent

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3	US 6221437 B1	7	☑			USPAT
4	US 6077412 A	25	☑			USPAT
5	US 5904827 A	10	☑			USPAT
6	US 5683564 A	11	☑			USPAT

US-PAT-NO: 6261435

DOCUMENT-IDENTIFIER: US 6261435 B1
See image for Certificate of Correction

TITLE: Plating method

---------- KWIC ---------

Brief Summary Text - BSTX (18):

In the plating method as described above, the treatment bath is vibrationally stirred by vibrating a vibration vane at an amplitude (width of vibration) range from 0.5 to 3.0 mm and at a vibrational frequency of 200 to 800 times per minute; the aeration is performed by using air bubbles generated by a diffuser pipe having a pore opening of 200 to 400 .mu.m; the plating target is swung at an amplitude (width of swing) of 10 to 100 mm and at a swing frequency of 10 to 30 times per minute; and, the plating target is vibrated at an amplitude of 0.5 to 1.0 mm and at a vibrational frequency of 100 to 300 times per minute.

Brief Summary Text - BSTX (20):

In the plating method as described above, the vibrationally stirring apparatus for the treatment bath preferably includes vibration generating means containing a vibration motor, vibrationally stirring means for vibrating at an amplitude of 0.5 to 3.0 mm and at a vibrational frequency of 200 to 800 times per minute a vibration vane which is fixed in one stage or in multistage to a vibrating bar which interlocks with the vibration generating means to vibrate in a treatment tank, an inverter for controlling the vibration motor to generate any low-frequency vibration at any frequency in the range from 10 to 500 Hz, preferably from 30 to 60 Hz, and more preferably from 30 to 40 Hz, and vibration stress dispersing means at a connection portion of the vibration generating means and the vibrationally stirring means.

Drawing Description Text - DRTX (8):

FIG. 7 is an enlarged partial cross-sectional view showing a manner of fixing vibration vanes to a vibration bar;

Detailed Description Text - DETX (10):

In FIGS. 4 and 5, on each vibrating bar 16, a spacer 30 is positioned between the neighboring vibration vanes 17 so that the vanes each held by a pair of vibration vane fixing members 18 are positioned at a certain interval.

Detailed Description Text - DETX (11):

The vibration vane 17 is preferably formed of thin metal, elastic synthetic resin, rubber or the like, and the thickness thereof may be set so that at least the tip portion of the vane plate shows a flutter phenomenon (as if it is corrugated) by the vertical oscillation of the vibration motor 4, whereby the

(12) **United States Patent**
Omasa

(10) Patent No.: **US 6,261,435 B1**
(45) Date of Patent: **Jul. 17, 2001**

(54) PLATING METHOD

(75) Inventor: Ryushin Omasa, Fujisawa (JP)

(73) Assignee: Nihon Techno Kabushiki Kaisha, Tokyo (JP)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 09/062,125

(22) Filed: Apr. 17, 1998

(30) Foreign Application Priority Data

Oct. 21, 1997 (JP) 9-306629

(51) Int. Cl.[7] C25D 5/34; C25D 5/00; C25D 5/20; B05D 3/04; B05D 3/12

(52) U.S. Cl. 205/205; 205/137; 205/148; 205/920; 427/96; 427/304; 427/346

(58) Field of Search 427/346, 304, 427/96; 205/146, 137, 205, 920

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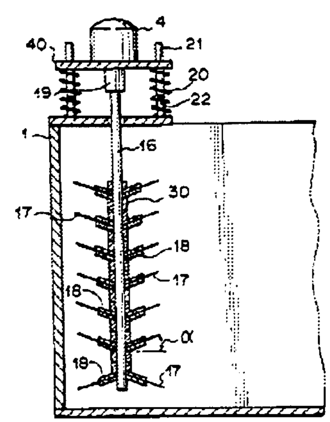
Primary Examiner—Edna Wong
(74) *Attorney, Agent, or Firm*—Pitney, Hardin, Kipp & Szuch LLP

(57) ABSTRACT

In a plating method for successively treating a plating target from a pre-treatment step until a plating treatment, (A) a vibrationally stirring apparatus for a treatment bath, (B) an aeration apparatus for the treatment bath, (C) an apparatus for swinging an electrode bar for suspending the plating target thereon, and (D) an apparatus for applying vibration to the electrode bar, are operated in a cleaning tank and at least one of an electroless plating tank and an electroplating tank used as a treatment tank in the pre-treatment step and the plating step.

13 Claims, 7 Drawing Sheets



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3	US 6221437 B1	7	USPAT
4	US 6077412 A	25	USPAT
5	US 5904827 A	10	USPAT
6	US 5683564 A	11	USPAT

US-PAT-NO: 6251250

DOCUMENT-IDENTIFIER: US 6251250 B1

TITLE: Method of and apparatus for controlling fluid flow and electric fields involved in the electroplating of substantially flat workpieces and the like and more generally controlling fluid flow in the processing of other work piece surfaces as well

---------- KWIC ---------

Detailed Description Text - DETX (5):

Referring to FIGS. 3 and 4, the construction of the cyclically reciprocating agitation rotor assembly 40 of the invention can be more readily seen. A plurality of spaced radially extending agitation blades or vanes 44 are attached to a rotor perimeter ring 46, FIG. 3. Each blade has a fluid flow window 48 cut into it that directs fluid flow to and from the wafer working surface 60 as the agitation rotor assembly rotates about its longitudinal rotation axis 50. A center flow hole 47 directs flow toward the center 104 of the wafer 60. Input fluid flow is channeled in a rotor blade flow channel 49 that extends from the rotor perimeter across the bottom of each blade 44 to connect with the center flow hole 47. The rotation axis 50 is disposed substantially perpendicular to the wafer working surface 60 and is aligned with the center of the wafer. Attached upon the rotor perimeter ring 46 is a rotor back iron 52 to which a plurality of circumferentially spaced magnets 54 are attached. The magnets 54 are suitably disposed with each of their magnetic field axes 56 oriented transversally, substantially perpendicular to the longitudinal rotation axis 50, and with the magnetic field direction alternating in north and south directions between adjacent magnets. The circumferential rotor back iron 52 and magnets 54 are coated with a suitable protective coating 58 to prevent their being corroded by the acidic plating solution or bath (24 in FIG. 2). A rubber-like material such as ethylene propylene provides an appropriate protective coating 58. Disposed around the rotor assembly 40 is a concentric outer stator 62, FIG. 3, with a plurality of successive circumferential teeth 64 and coil windings 66, defining an annular space between the ring of rotor magnets and the coaxially surrounding stator teeth that may be referred to as a motor air gap 68. In combination, the rotor back iron 52, magnets 54, stator 62, windings 66 and motor air gap 68 form a permanent magnet DC motor.

Claims Text - CLTX (42):

42. In a wet processing system in which processing fluid flows against a fixed workpiece surface contained within a cylindrical fluid processing chamber, a method of improving the control of fluid flow and the uniformity of the processing of the workpiece, that comprises, agitating the fluid by internally cyclically reciprocally rotating spaced radial vanes disposed within the fluid and extending from the chamber to the inner walls thereof.

Claims Text - CLTX (43):

(12) **United States Patent**
Kelgler

(10) Patent No.: **US 6,251,250 B1**
(45) Date of Patent: **Jun. 26, 2001**

(54) **METHOD OF AND APPARATUS FOR CONTROLLING FLUID FLOW AND ELECTRIC FIELDS INVOLVED IN THE ELECTROPLATING OF SUBSTANTIALLY FLAT WORKPIECES AND THE LIKE AND MORE GENERALLY CONTROLLING FLUID FLOW IN THE PROCESSING OF OTHER WORK PIECE SURFACES AS WELL**

(76) Inventor: Arthur Keigler, 20 Pine Plain Rd., Wellesley, MA (US) 02461

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 09/390,110

(22) Filed: Sep. 3, 1999

(51) Int. Cl.^{7} C25D 5/08; C25D 5/22; C25D 21/10; C25D 7/12

(52) U.S. Cl. ..... 205/89; 205/96; 205/133; 205/148; 205/157; 204/224 R; 204/273; 204/230.2; 204/275.1

(58) Field of Search ..... 205/96, 148, 261, 205/101, 157, 89, 133; 204/242, 275.1, 269, 273, 222, 224 R, 230.2

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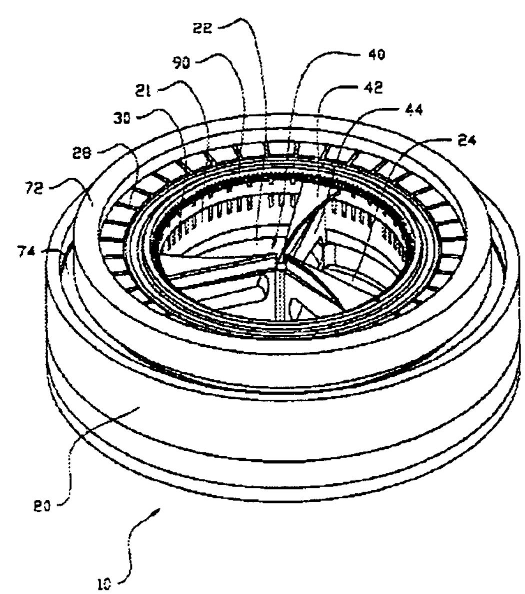
Primary Examiner—Kathryn Gorgos
Assistant Examiner—Michael J Feely
(74) Attorney, Agent, or Firm—Rines and Rines

(57) **ABSTRACT**

A novel method and apparatus of wet processing workpieces, such as electroplating semiconductor wafers and the like, that incorporates reciprocating processing fluid agitation to control fluid flow at the workpiece, and where electric fields are involved as in such electroplating, controlling the electric field distribution.

51 Claims, 7 Drawing Sheets



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3	US 6221437 B1	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
4	US 6077412 A	25	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
5	US 5904827 A	10	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
6	US 5683564 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 5683564

DOCUMENT-IDENTIFIER: US 5683564 A
See image for Certificate of Correction

TITLE: Plating cell and plating method with fluid wiper

---------- KWIC ---------

Brief Summary Text - BSTX (18):

According to an aspect of the present invention, in an electroplating cell a planar face of a substrate is plated with a metal layer. A plating bath contains an electrolyte in which the substrate is immersed. A sparger introduces the electrolyte into the bath. An anode chamber contains an anode basket holding a quantity of metal that is consumed during plating. A weir separates the anode chamber from the bath and permits the electrolyte to spill over from the bath into the anode chamber. The weir can have a semipermeable membrane wall that permits metal ions to pass through from the anode chamber into said plating bath, but blocks the flow of the electrolyte and any entrained particulates. A drain outlet carries electrolyte and any entrained particulate matter from the anode chamber. Also, conditioning and handling equipment coupled between the drain outlet and the sparger removes any particulate matter from the electrolyte and returns the electrolyte through a return conduit to the sparger. A rotary blade or wiper is positioned in the plating bath between the semipermeable membrane wall and the substrate, and has an edge disposed a predetermined distance from the planar face of the substrate. This distance is below about one-half inch, and is preferably about three-eighths inch. Preferably, the blade or wiper is pitched in the direction such that the rotating wiper tends to pull the electrolyte, plus any hydrogen bubbles, away from the substrate. The rotary wiper is most preferably fluid powered, and is coupled to the electrolyte return conduit to receive a flow of the electrolyte as motive power therefor. In several preferred embodiments, the fluid powered wiper includes an annular turbine having a generally circular opening therethrough, with the annular turbine being mounted in a circular mount therefor that is disposed in the plating bath. The circular opening is in registry with the substrate face that is to be plated. The blade is mounted on the annular turbine to extend radially towards a center of said circular opening. The annular turbine can have vanes disposed around its periphery, and the circular mount can have an annular recess that covers the periphery of the turbine and around which the vanes travel. A conduit is provided from the return conduit to the annular recess to propel the turbine and vane . As the same filtered and conditioned electrolyte that is fed through the sparger into the plating bath is also used to power the turbine, the leakage from this turbine will not in any way contaminate or dilute the electrolyte in the plating bath. The same materials that are used in the walls of the plating cell, e.g., a high quality polypropylene or PFA Teflon, are also used for the rotary blade, turbine, and mount. The annular turbine can be supported for rotation by rollers (formed of the same or a compatible plastic resin) mounted on the support for the annular turbine. This avoids the need for any bearings or metallic parts.

Detailed Description Text - DETX (7):

As shown in FIG. 3 a rotary wiper or blade unit 70 is fitted into the weir

United States Patent [19]

Reynolds

[11] Patent Number: 5,683,564

[45] Date of Patent: Nov. 4, 1997

[54] PLATING CELL AND PLATING METHOD WITH FLUID WIPER

[75] Inventor: H. Vincent Reynolds, Marcellus, N.Y.

[73] Assignee: Reynolds Tech Fabricators Inc., E. Syracuse, N.Y.

[21] Appl. No.: 731,508

[22] Filed: Oct. 15, 1996

[51] Int. Cl.[6] C25D 17/02; C25D 21/10

[52] U.S. Cl. 205/68; 204/212; 204/238; 204/264; 204/273; 205/70; 205/99; 205/148

[58] Field of Search .... 205/68, 70, 99, 205/101, 148; 204/212, 224 R, 238, 264, 273

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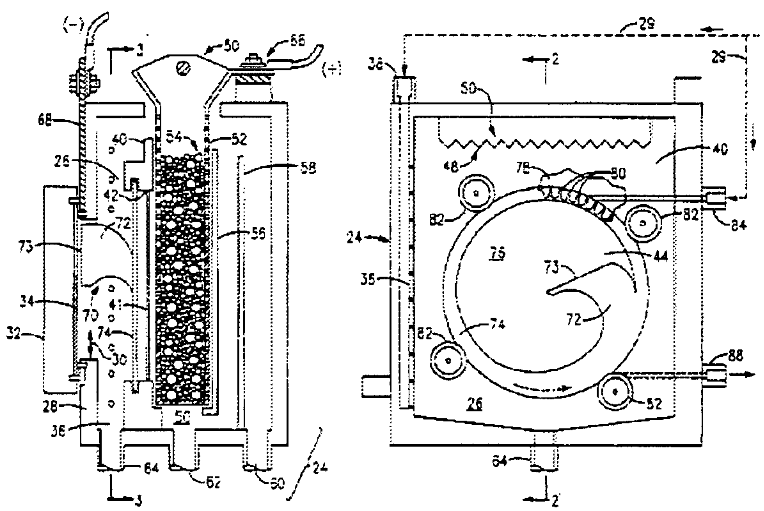
Primary Examiner—Kathryn L. Gorgos
Assistant Examiner—William T. Leader
Attorney, Agent, or Firm—Trapani & Molldrem

[57] ABSTRACT

A plating cell for plating a flat substrate, for example, a stamper for a high-density compact disk recording, employs a sparger to introduce a flow of electrolyte across the surface of the substrate to be plated. A fluid-powered rotary blade or wiper within the cathode chamber has a rotary blade with an edge spaced a small distance, preferably about three-eighths inch, from the substrate, and an annular turbine which rotates under a flow of the electrolytic fluid that is also being fed to the sparger. The rotary wiper is run at a speed between about 35 and 80 rpm and draws the electrolyte away from the substrate. This helps remove hydrogen bubble that form during electroplating. A semipermeable weir separates the cathode chamber from an anode chamber that contains an anode basket that is filled with plating material. The plating cell is provided with a backwash flow regime so that impurities and inclusions from the anode chamber are kept out of the plating bath.

22 Claims, 4 Drawing Sheets



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19	US 3933601 A	5	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT

Brief Summary Text - BSTX (54):

Next, in order to attain the afore-mentioned effect, particularly by dispersing solid particles in the fused electrolyte, the electrolyte must have a sufficient relative velocity at the deposit surface. It will be apparent that a sufficient relative velocity can be produced near the deposit surface by the above described mechanical fluid transport means with the cathode electrode being motionless or slowly revolving or shifting, and/or the aforesaid sufficient relative velocity can be produced by violently vibrating or rortating the cathode electrode as shown in the Examples.

Brief Summary Text - BSTX (59):

The average current density utilized during the electrodeposition of the present invention can be substantially varied, and to a large extent, is influenced by the size of the cathode selected. While definitive values cannot be set due to the wide variation in cathode size which can be utilized in the present invention, the general rule to be followed is that larger the cathode the higher the current density used, and the smaller the cathode the lower the current density used. Optimum current density is generally determined in an empirical fashion, i.e., as is conventional in the art a few process runs are conducted at varying average current densities until the current density which provides the best product is determined. For example, using a cathode with a diameter of 32 mm the maximum average current density may be bout 30 A/dm.sup.2, whereas using a vibrating cathode of a larger size as would commonly be used on an industrial scale an average current density of 80 A/dm.sup.2 or higher is realistic for the electrodeposition of titanium metal by the process of the present invention is an agitated bath, for example.

Detailed Description Text - DETX (111):

The cathode electrode was vibrated with a vibration period of 400 cycles/min, amplitude 36 mm and vibration direction 45.degree. to the cathode flat surface.

Detailed Description Text - DETX (118):

The electrolyte of Reference 3 had further added thereto metallic titanium and TiCl.sub.3 according to the previously described method of adding the titanium salt component, and the temperature of this electrolyte was increased to a temperature sufficient to melt all components of the fused electrolyte. Thereafter, the temperature was lowered to 400.degree. C to obtain an electrolyte (containing crystallized solid particles having a size estimated to be on the order of the solid particles added exterior of the system in Examples 1 and 2), comprising 19.2 weight % of TiCl.sub.2 and 7.0 weight % of TiCl.sub.3 near the cathode position about 3 cm under the electrolyte surface. This electrolyte was used in this example and the same vibration of the cathode electrode as in Reference Example 3 was carried out.

Detailed Description Text - DETX (135):

The cathode electrode was vibrated with a vibrator period 400 cycle/min.(0.15 seconds) and a 30mm amplitude. Vibration direction: 45.degree. toward the cathode sufrace.

1

ELECTRODEPOSITION PROCESS

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a continuous electrodeposition process wherein solid particles are dispersed in a fused-salt electrolyte to obtain an electrodeposited metal or alloy which is extremely flat and free of flaws on the surface of cathode immersed in the electrolyte.

2. Description of the Prior Art

In the prior art, when a desired metal or alloy is to be electrodeposited as a solid by fused-salt electrolysis, the electrodeposited metal is often in the form of a powder, aggregate crystal, dendrite or sponge. When the desired metal or alloy electrodeposited in the above form is recovered, a great amount of electrolyte is inevitably lost during separating the metal or alloy from the electrolyte. Further, when the desired metal or alloy has high reactivity with oxygen, nitrogen and the like, the high surface area caused by the aforesaid form of the electrodeposited metal is subject to contamination by reaction with such different elements. Thus, after treatments of the deposited metal are usually accompanied by many difficulties.

Further, even when the metal or alloy can be electrodeposited as a relatively homogeneous film, conditions which permit such to be obtained are very narrow, e.g., a small allowable limit for the cathode current density and so on. Accordingly, undesirable restrictions have been imposed on processes for producing such film materials.

U.S. Pat. No. 3,662,047 issued May 9, 1972, in the name of Tokumoto et al discloses a method for the electrodeposition of titanium or a titanium alloy. A fused salt electrolytic bath containing a mixture of chloride salts of barium, magnesium, sodium and calcium having a freezing point of less than 600° C and titanium dichloride, and, if desired, a source of a suitable metal alloy, is electrolyzed. The electrolytic bath is maintained at a temperature of 400° to 580° C and under such conditions as will maintain the molar ratio of titanium trichloride to titanium dichloride at less than 0.5 in the vicinity of the electrode to be electrodeposited.

SUMMARY OF THE INVENTION

Accordingly, a main object of this invention is to provide an electrodeposition process which improves upon the above identified processes and/or is free from the above drawbacks.

Another object of this invention is to provide an electrodeposition process wherein a fused electrolyte is used, even with a metal whose electrodeposition as a relatively homogeneous, dense film is considered difficult, whereby a desired metal or alloy can be electrodeposited with its surface being kept flat on an electrode under operating conditions which are industrially useful, whereby a relatively homogeneous, dense film of electrodeposited metal or alloy having a predetermined thickness and high flatness can be obtained.

The above and other objects of the present invention are obtained by the presence of dispersed particles in the electrodeposition bath during the electrodeposition. While the particles may be added per se to the electrodeposition bath or formed in situ, in both instances the agitation effect of the dispersed particles permits one to obtain an electrodeposited metal or alloy layer which is extremely flat and free of flaws at the surface of the cathode in the electrodeposition bath.

The above and other objects and features of the invention will appear more fully hereinafter from a consideration of the following description accompanied with preferred embodiments.

2

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Before the description of the present invention, a fused electrolyte as is normally used in a fused salt electrolysis will be described.

A fused electrolyte or electrolytic bath capable of dissolving a desired metal or alloy deposited by electrolysis comprises a higher valent compound of the desired metal in the fused electrolyte which reacts with electrodeposited metal to produce a lower valent compound of the desired metal, thus providing the deposited metal corroded and dissolved therein. One example thereof is a fused electrolyte where an electrodeposited metal is corroded and dissolved in the electrolyte by a disproportional reaction or disproportionation. A disproportionation of this kind will be described with reference to the electrodeposition of titanium, by way of example. As will be appreciated by one skilled in the art, in the electrodeposition of titanium from a fused-salt electrolyte, the starting material actually used in $TiCl_4$, which is reduced into $TiCl_3$ and $TiCl_2$ in a conventional manner. Since in the electrodeposition of titanium using a fused-salt delectrolyte this step inherently occurs, in the following examples the starting portions of $TiCl_3$ and $TiCl_2$ are given for purposes of convenience, as such terminology is more conventionally used in the art.

Between a higher valent compound of titanium such as $TiCl_4$, K_2TiCl_6, $BaTiCl_6$ or the like contained in a fused electrolyte and metallic titanium deposited by electrolysis, the following disproportionation exists at least in the temperature range of about 400° to 600° C.

$$Ti + 2TiCl_3 \rightarrow 3TiCl_2 \quad (1)$$

$$Ti + K_2TiCl_6 \rightarrow 2KCl + 2TiCl_2 \quad (2)$$

$$Ti + BaTiCl_6 \rightarrow BaCl_2 + 2TiCl_2 \quad (3)$$

When a trivalent complex of titanium such as Cs_3TiCl_6 is present in the electrolyte, a similar disproportionation is also seen. In either case, however, the metallic titanium deposited by the electrolysis reacts with a higher valent titanium compound, such as a trivalent or tetravalent compound, in the electrolyte to produce a lower valent titanium compound, such as a divalent compound, which is dissolved in the electrolyte.

Another example of a fused electrolyte capable of dissolving a desired metal or alloy deposited by electrolysis is one where the metal or alloy can be subjected to anodic dissolution in the fused electrolyte.

A further example of a fused electrolyte capable of dissolving a desired metal or alloy deposited by electrolysis is one where the desired metal is locally dissolved in the electrolyte by the electromotive force of a concentration cell based upon the concentration difference of desired metal ions locally produced on the surface of the electrodeposit (hereinafter referred to as a deposit surface).

5/2003 SN 09/864,650 47

EAST Browser - L7: (22) 1 and 6 | US 5830334 A | Tag: S | Doc: 5/22 (SORTED)

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3	US 6036837 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
4	US 5911863 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
5	US 5830334 A	10	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
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US-PAT-NO: 5830334

DOCUMENT-IDENTIFIER: US 5830334 A

TITLE: Nozzle for fast plating with plating solution jetting and suctioning functions

---------- KWIC ---------

Brief Summary Text - BSTX (6):

It is also well known in the art to increase the plating speed by moving the cathode and at the same time agitating the plating solution in the vicinity of the cathode surface. The agitation of plating solution to this end is done with various means. Among these means are one utilizing air agitation which is more intense than the usual agitation, one in which the entire plating solution is agitated with a propeller, one utilizing low frequency vibrations, one utilizing ultrasonic waves, and one utilizing pump recirculation of the plating solution to be jetted from a nozzle. These agitating means, however, have their own drawbacks. With air agitation, the current density is increased only up to a level corresponding to a couple of times the usual plating speed. Besides, it is extremely difficult in this case to obtain a uniform plating film thickness. In the case of propeller agitation of the entire plating solution, it is difficult to obtain uniform agitation with respect to a product. For this reason, this agitating means is hardly used. Utilizing low frequency vibrations is not very effective for increasing the plating speed; it permits only a plating speed increase comparable to that in the case of the air agitation. Utilizing ultrasonic waves readily results in a coarsened surface of material. Besides, the frequency used varies with the kind of plating solution. At present, therefore, this agitation method is hardly used.

Current US Cross Reference Classification - CCXR (4):

205/148

United States Patent [19]

Kobayashi

[11] **Patent Number: 5,830,334**

[45] **Date of Patent: Nov. 3, 1998**

[54] **NOZZLE FOR FAST PLATING WITH PLATING SOLUTION JETTING AND SUCTIONING FUNCTIONS**

[76] Inventor: Hideyuki Kobayashi, 1-11-11, Omiya-cho, Mishima-shi, Shizuoka-ken, Japan

[21] Appl. No.: 746,290

[22] Filed: Nov. 7, 1996

[51] Int. Cl.[6] C25D 17/00

[52] U.S. Cl. .... 204/224 R; 204/275; 204/279; 205/133; 205/148; 239/104

[58] Field of Search .... 205/133, 148, 205/670; 204/224 R, 224 M, 275, 279; 239/104; 118/300

[56] References Cited

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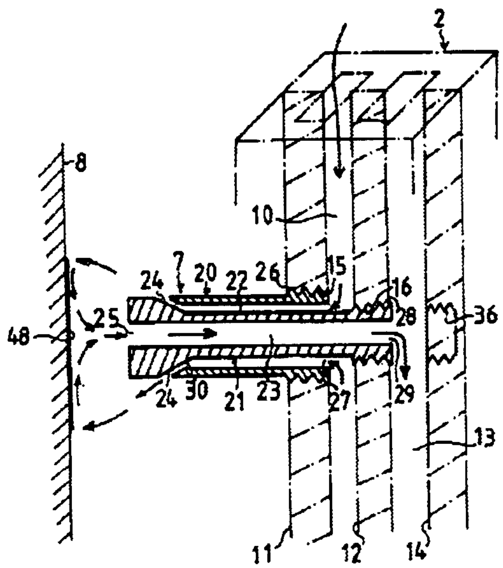
5,382,343	1/1995	Zwerner	204/224 R
5,397,453	3/1995	Imori	205/123

Primary Examiner—Kathryn L. Gorgos
Assistant Examiner—William T. Leader
Attorney, Agent, or Firm—Wenderoth, Lind & Ponack

[57] **ABSTRACT**

A nozzle for fast plating with plating solution jetting and sucking functions includes an outer cylindrical member and an inner cylindrical member, a plating solution issuing passage being defined between the outer and inner cylindrical members, and the inner space in the inner cylindrical member constituting a plating solution sucking passage. The inner cylindrical member has a front end flared portion having a forwardly flared surface acting to diffuse the solution. The outer cylindrical member has a front open end defining a gap with respect to the flared surface, the gap constituting a jetting port of the plating solution jetting passage. Plating solution supplied from a supply opening of the plating solution jetting passage is jetted from the jetting port toward a workpiece to be plated, the jetted plating solution is discharged from a rear end discharge opening of the plating solution sucking passage.

12 Claims, 4 Drawing Sheets



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  - L3: (6) 11 and 12
  - L4: (70421) vibrate or vibrates or vibrated or vibrating
  - L5: (184265) vibration or vibrations or vibrational
  - L6: (211397) 14 or 15
  - L7: (22) 11 and 16
  - L8: (122) (205/104).CCLS.
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	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor
1	US 6387243 B1	20020514	12	Separation of metal ions absorbed on a resin and	205/687	204/228.6; 204/229.4;		Gasser, Gerard
2	US 6375821 B1	20020423		Method for coating electrically conductive	205/83	204/472; 204/499;		Jerome, Robert et al.
3	US 6129830 A	20001010		Process for the electrolytic deposition of copper layers	205/104	205/125		Senge, Gerd et al.
4	US 6099711 A	20000808		Process for the electrolytic deposition of metal layers	205/101	205/103; 205/104;		Dahms, Wolfgang et al.
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6	US 5705230 A	19980106		Method for filling small holes or covering small	427/438	205/104; 205/191;		Matanabe, Toru et al.
7	US 5605615 A	19970225		Method and apparatus for plating metals	205/83	204/229.2; 204/229.7;		Goolsby, Peter G. et al.
8	US 4364802 A	19821221		Scanning electrode vibration electrodeposition method	205/104	205/117; 205/118;		Inoue, Kiyoshi
9	US 4101386 A	19780718		Methods of coating and surface finishing articles	205/211	205/103; 205/104;		Dotzer, Richard et al.
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11	US 3994785 A	19761130		Electrolytic methods for production of high density	205/74	204/223; 204/DIG.9;		Rippere, Ralph E.

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EAST Browser - L10: (11) 8 and 6 | US 6129830 A | Tag: S | Doc: 3/11 (SORTED) | Format : KWIC

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3	US 6129830 A	6	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
4	US 6099711 A	13	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT
5	US 5911863 A	7	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT
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7	US 5605615 A	10	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT

US-PAT-NO: 6129830

DOCUMENT-IDENTIFIER: US 6129830 A

TITLE: Process for the electrolytic deposition of copper layers

---------- KWIC ---------

Brief Summary Text - BSTX (34):

The deposition bath is moved by a strong incident flow and possibly by blowing in clean air to strongly agitate the bath surface. This maximizes the transport of materials to the cathodes and anodes so that higher current densities are possible. Moving the cathodes also helps transport substances to the respective surfaces. The higher convection and electrode movement produces a constant, diffusion-controlled deposition. The substrates can move horizontally, vertically and/or by vibration . It is particularly effective to both move the substrates and blow air into the deposition bath.

Current US Original Classification - CCOR (1):

205/104

United States Patent [19]

Senge et al.

[11] Patent Number: 6,129,830

[45] Date of Patent: Oct. 10, 2000

[54] PROCESS FOR THE ELECTROLYTIC DEPOSITION OF COPPER LAYERS

[75] Inventors: Gerd Senge; Wolfgang Dahms, both of Berlin, Germany

[73] Assignee: Atotech Deutschland GmbH, Germany

[21] Appl. No.: 09/319,423

[22] PCT Filed: Dec. 4, 1997

[86] PCT No.: PCT/EP97/06786

§ 371 Date: Jun. 2, 1999

§ 102(e) Date: Jun. 2, 1999

[87] PCT Pub. No.: WO98/26114

PCT Pub. Date: Jun. 18, 1998

[30] Foreign Application Priority Data

Dec. 13, 1996 [DE] Germany 196 53 681

[51] Int. Cl.⁷ C25D 5/18; C25D 5/02

[52] U.S. Cl. 205/104; 205/125

[58] Field of Search 205/104, 125

[56] References Cited

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215 589 11/1984 Germany .
261 613 11/1988 Germany .
43 44 387 6/1995 Germany .
07242396 8/1995 Japan .
662217 12/1991 United Kingdom .
PCT/GB89/00042 1/1989 WIPO .

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"Pulse Plating of Copper for Printed Circuit Board Technology", Metal Finishing Apr. 21–21, 1991, M.R. Kalantary and D.R. Gabe, Loughborough University of Technology, Leicestershire, England and M. Goodenough, Lea Ronal (UK) Ltd. Buxton, England. See p. 3, paragraph 2 of the specification.

Primary Examiner—Kathryn Gorgos
Assistant Examiner—Erica Smith-Hicks
Attorney, Agent, or Firm—Paul & Paul

[57] ABSTRACT

A process with the following process steps is used to electrolytically deposit copper layers, especially on printed circuit boards. An electrically-conductive substrate and anodes that decompose upon electrolytic deposition are brought into contact with a deposition bath. The deposition bath contains copper ions, compounds that increase the electrical conductivity of the deposition bath, additives to influence the material properties of the copper layers, additional compounds of an electrochemically reversible redox system, and solvents or solvent mixtures. The substrate and the electrodes are connected to a power supply. The copper layers are deposited on the substrate using a pulsed current or a pulsed voltage process. When this process is used, metal layers with favorable visual and mechanical material properties are deposited after a brief bath preparation time.

13 Claims, No Drawings

5/2003 SN 09/864,650 line 10

EAST Browser - L10 (11) 8 and 6 | US 6129830 A | Tag: S | Doc: 3/11 (SORTED) | Format : KWIC

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3	US 6129830 A	6		USPAT
4	US 6099711 A	13		USPAT
5	US 5911863 A	7		USPAT
6	US 5705230 A	9		USPAT
7	US 5605615 A	10		USPAT

US-PAT-NO: 6129830

DOCUMENT-IDENTIFIER: US 6129830 A

TITLE: Process for the electrolytic deposition of copper layers

---------- KWIC ---------

Brief Summary Text - BSTX (34):

The deposition bath is moved by a strong incident flow and possibly by blowing in clean air to strongly agitate the bath surface. This maximizes the transport of materials to the cathodes and anodes so that higher current densities are possible. Moving the cathodes also helps transport substances to the respective surfaces. The higher convection and electrode movement produces a constant, diffusion-controlled deposition. The substrates can move horizontally, vertically and/or by vibration . It is particularly effective to both move the substrates and blow air into the deposition bath.

Current US Original Classification - CCOR (1):

205/104

United States Patent [19]

Senge et al.

[11] Patent Number: 6,129,830

[45] Date of Patent: Oct. 10, 2000

[54] PROCESS FOR THE ELECTROLYTIC DEPOSITION OF COPPER LAYERS

[75] Inventors: Gerd Senge; Wolfgang Dahms, both of Berlin, Germany

[73] Assignee: Atotech Deutschland GmbH, Germany

[21] Appl. No.: 09/319,423

[22] PCT Filed: Dec. 4, 1997

[86] PCT No.: PCT/EP97/06786

§ 371 Date: Jun. 2, 1999

§ 102(e) Date: Jun. 2, 1999

[87] PCT Pub. No.: WO98/26114

PCT Pub. Date: Jun. 18, 1998

[30] Foreign Application Priority Data

Dec. 13, 1996 [DE] Germany 196 53 681

[51] Int. Cl.[7] C25D 5/18; C25D 5/02

[52] U.S. Cl. 205/104; 205/125

[58] Field of Search 205/104, 125

[56] References Cited

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215 589	11/1984	Germany .
261 613	11/1988	Germany .
43 44 387	6/1995	Germany .
07242396	8/1995	Japan .
662217	12/1991	United Kingdom .
PCT/GB89/00042	1/1989	WIPO .

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"Pulse Plating of Copper for Printed Circuit Board Technology", Metal Finishing Apr. 21–21, 1991. M.R. Kalantary and D.R. Gabe, Loughborough University of Technology, Leicestershire, England and M. Goodenough, Lea Ronal (UK) Ltd. Buxton, England. See p. 3, paragraph 2 of the specification.

Primary Examiner—Kathryn Gorgos
Assistant Examiner—Erica Smith-Hicks
Attorney, Agent, or Firm—Paul & Paul

[57] ABSTRACT

A process with the following process steps is used to electrolytically deposit copper layers, especially on printed circuit boards. An electrically-conductive substrate and anodes that decompose upon electrolytic deposition are brought into contact with a deposition bath. The deposition bath contains copper ions, compounds that increase the electrical conductivity of the deposition bath, additives to influence the material properties of the copper layers, additional compounds of an electrochemically reversible redox system, and solvents or solvent mixtures. The substrate and the electrodes are connected to a power supply. The copper layers are deposited on the substrate using a pulsed current or a pulsed voltage process. When this process is used, metal layers with favorable visual and mechanical material properties are deposited after a brief bath preparation time.

13 Claims, No Drawings

5/2003 SN 09/864,650 line 10

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7	US 5605615 A	10						☑					USPAT

TITLE: Method for filling small holes or covering small recesses in the surface of substrates

---------- KWIC ---------

Drawing Description Text - DRTX (2):

FIG. 1 shows a setup for implementing the method of the present invention with a vibration energy being applied to a substrate;

Drawing Description Text - DRTX (7):

FIG. 6 shows the result of covering small recesses in the surface of a substrate by the method of the present invention, with a vibrating energy being applied at 50 Hz.

Detailed Description Text - DETX (3):

In the step of deposition, a controlled varying voltage and/or an energy such as low-frequency, high-frequency or ultrasonic vibrations or a light beam is preferably applied to the substrate. The applicable varying voltage includes voltages of pulsed waveforms such as square wave, trapezoidal wave, triangular wave, sinusoidal wave and random wave. The voltages of such pulsed waveforms may have both positive and negative polarities or they may have only negative polarity. The repetitive voltage components may be zero at periodic times; alternatively, a d.c. component may be added to a periodic wave. The value of the voltage to be applied is preferably such that a maximum absolute value of current density on the surface of the substrate Is in the range of from 10.sup.-3 to 1 A/cm.sup.2. The low-frequency, high-frequency or ultrasonic vibrations are preferably such that their frequency is in the range of from 45 Hz to 2.5 GHz.

Detailed Description Text - DETX (4):

Application of voltages or vibrations may be performed with any known apparatus used In the art. A setup for implementing the method of the present invention with a vibration energy being applied is shown in FIG. 1, and a setup for implementing the method of the present Invention with a light energy being applied is shown in FIG. 2. If desired, the small holes and recesses in the surface of a substrate may be first filled or covered with a pure metal or an alloy by the conventional means and, thereafter, the same or different method is applied to have another pure metal or alloy be deposited over the filling or covering layer of the first pure metal or alloy. A specific embodiment of this approach is as follows: the small holes or recesses in the surface of a substrate are first filled or covered with Ag or an Ag alloy or Cu or a Cu alloy and, then, Ni or a Ni alloy such as Ni--B is deposited over the filling or covering layer. This method enables either Implementation of the hot post-treatment which is effective for the process of semiconductor fabrication (since heat resistance is Improved) or prevention of the occurrence of a deleterious surface reaction. It may sometimes become possible to suppress neighboring elements in the filling or covering layer from moving around or penetrating through said layer on account of heat diffusion (which can lead to materials deterioration).

US005705230A

United States Patent [19]

Matanabe et al.

[11] Patent Number: 5,705,230

[45] Date of Patent: Jan. 6, 1998

[54] METHOD FOR FILLING SMALL HOLES OR COVERING SMALL RECESSES IN THE SURFACE OF SUBSTRATES

[75] Inventors: Toru Matanabe, Hopewell Junction, N.Y.; Hirokazu Ezawa, Tokyo, Japan; Masahiro Miyata, Chiba-ken, Japan; Yukio Ikeda, Tokyo, Japan; Manabu Tsujimura, Kanagawa-ken, Japan; Hiroaki Inoue, Kanagawa-ken, Japan; Takeyuki Odaira, Kanagawa-ken, Japan; Naoaki Ogure, Tokyo, Japan

[73] Assignee: Ebara Corporation, Tokyo, Japan

[21] Appl. No.: 324,460

[22] Filed: Oct. 17, 1994

Related U.S. Application Data

[63] Continuation of Ser. No. 32,657, Mar. 17, 1993, abandoned.

[30] Foreign Application Priority Data

Mar. 17, 1992 [JP] Japan 4-091778

[51] Int. Cl.⁶ B05D 1/18

[52] U.S. Cl. 427/438; 205/91; 205/104; 205/191

[58] Field of Search 205/91, 118, 161, 205/191; 427/437, 438, 443.1, 595

[56] References Cited

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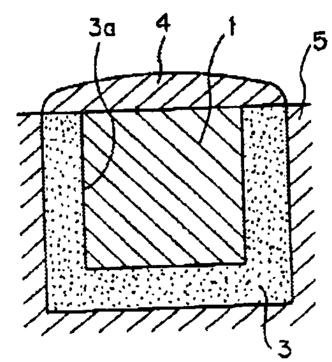
Primary Examiner—John Niebling
Assistant Examiner—Brendan Mee
Attorney, Agent, or Firm—Oblon, Spivak, McClelland, Maier & Neustadt, P.C.

[57] ABSTRACT

The improved method comprises contacting a substrate 5 at least once by a liquid containing the elements that compose a pure metal or an alloy with which the small holes or recesses 3a in the substrate 5 are to be filled or covered, whereby the liquid wets the inner surfaces of said small holes or recesses 3a while, at the same time, said pure metal or said alloy is deposited on the surface of said substrate 5. The method is capable of filling small holes or covering small recesses in the surface of the substrate 5 with improved efficiency while, at the same time, it improves the heat resistance and materials stability of the part that contains the formed filling or covering layer.

3 Claims, 4 Drawing Sheets



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5	US 6077412 A	25	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT
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7	US 5730856 A	22	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT

US-PAT-NO: 6322689

DOCUMENT-IDENTIFIER: US 6322689 B1

TITLE: Anodizing method and apparatus for performing the same

---------- KWIC ---------

Brief Summary Text - BSTX (13):

(a) a step of vibrationally fluidly stirring the treatment bath, wherein the treatment bath is vibrationally fluidly stirred by vibrating a vibration vane at an amplitude from 0.5 to 3.0 mm and at a vibrational frequency of 200 to 800 times per minute; and

Brief Summary Text - BSTX (19):

(A) a vibrationally fluidly stirring apparatus for the treatment bath, which comprises a vibration generating means containing a vibration motor, a vibrationally fluidly stirring means for vibrating a vibration vane at an amplitude of 0.5 to 3.0 mm and at a vibrational frequency of 200 to 800 times per minute to generate vibrational flow in the treatment bath, the vibration vane being fixed in one stage or in multistage to a vibrating bar which vibrates in the treatment bath interlockingly with the vibration generating means, and a vibration stress dispersing means at a connection portion of the vibration generating means and the vibrationally fluidly stirring means; and

Brief Summary Text - BSTX (44):

In the method of the present invention, a post-treatment process may be performed as usual after the anodizing treatment process. Example of the post-treatment process comprises a sealing step for treating the porous surface of the metal body. The sealing step can be performed by steam sealing, metal salt sealing, electrodeposition sealing, dye sealing, or pigment sealing, or, the combination thereof.

Drawing Description Text - DRTX (11):

FIG. 10 is an enlarged cross-sectional view showing a manner of fixing vibration vanes to a vibration bar;

Detailed Description Text - DETX (3):

In FIGS. 1 to 3, the aeration apparatus (B) includes three diffusing pipes 12 disposed on the bottom plate of the treatment tank 1, and compressed air inlet ports 10 through which compressed air is fed to the diffusing pipes 12. Reference numeral 4 denotes a vibration motor, 16 a vibrating bar, 17 a vibration vane. These are parts of the vibrationally fluidly stirring apparatus (A).

Detailed Description Text - DETX (5):

(12) United States Patent
Omasa

(10) Patent No.: US 6,322,689 B1
(45) Date of Patent: *Nov. 27, 2001

(54) ANODIZING METHOD AND APPARATUS FOR PERFORMING THE SAME

(75) Inventor: Ryushin Omasa, Fujisawa (JP)

(73) Assignee: Japan Techno Co., Ltd. (JP)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: 09/536,844

(22) Filed: Mar. 28, 2000

(30) Foreign Application Priority Data

Apr. 2, 1999 (JP) 11-096832

(51) Int. Cl.[7] C25D 17/00

(52) U.S. Cl. 205/324; 205/316; 205/323; 204/222; 204/273

(58) Field of Search 204/222, 273; 205/671, 324, 316–333

(56) References Cited

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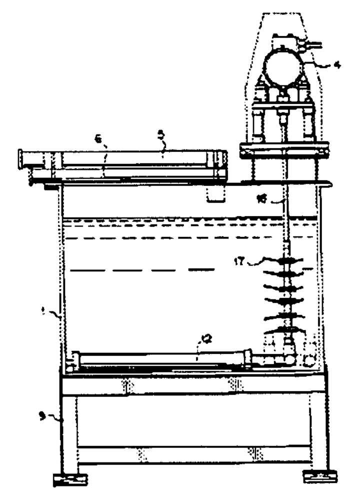
Primary Examiner—Robert Dawson
Assistant Examiner—Michael J Feely
(74) Attorney, Agent, or Firm—Fitzey, Hardin, Kipp & Szuch, LLP

(57) ABSTRACT

An anodizing method of a metal body which includes an anodizing treatment process performed while vibrationally fluidly stirring and aerating a treatment bath in which the metal body is vibrated and swung and an apparatus for performing the same.

19 Claims, 14 Drawing Sheets



5/2003 SN 09/864,650 line 16

EAST Browser - L16 (16) 15 and 2 | US 5730856 A | Tag: S | Doc: 7/16 (SORTED) | Format : KWIC

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	Document ID	Pages	Kind Codes	Source
2	US 6261433 B1	23		USPAT
3	US 6251250 B1	17		USPAT
4	US 6123815 A	18		USPAT
5	US 6077412 A	25		USPAT
6	US 5932077 A	8		USPAT
7	US 5730856 A	22		USPAT
8	US 5683564 A	11		USPAT

US-PAT-NO: 5730856

DOCUMENT-IDENTIFIER: US 5730856 A

TITLE: Method for treating waste liquid with electrolytic oxidation and apparatus for carrying out the same

---------- KWIC ---------

Abstract Text - ABTX (1):

An electroless nickel plating waste liquid (4) accommodated in an electrolytic oxidation tank (2) is treated by electrolytic oxidation with vibrating and fluidizing the waste liquid by means of an oscillating stirrer (10) to recover nickel by the electrolytic oxidation as well as recover the waste liquid in which an active component for fertilizer including phosphorus remains as a fertilizer solution. When the waste liquid is neutralized by a neutralizer including an active component for fertilizer such as potassium, nitrogen, etc., the fertilizer solution having higher content of the active component is obtained. The oscillating stirrer comprises an oscillation generator including a 25 to 500 Hz oscillating motor (18), an oscillating rod (22) connected to the oscillation generator, and multistage oscillating vanes (38) attached to the oscillating rod, and the oscillating vane is such that a tip end portion of the vane is fluttered when an oscillation is transmitted from the oscillating rod to the vane .

Brief Summary Text - BSTX (15):

the oscillating stirrer comprising an oscillation generator, an oscillation transmitter connected to the oscillation generator, and one-stage or multistage oscillating vane attached to the oscillation transmitter, the oscillating vane being such that a tip end portion thereof is fluttered when an oscillation is transmitted from the oscillation transmitter to the vane .

Brief Summary Text - BSTX (23):

Preferably, the oscillating stirrer comprises an oscillation generator, an oscillation transmitter connected to the oscillation generator, and one-stage or multistage oscillating vane attached to the oscillation transmitter, the oscillating vane being such that a tip end portion thereof is fluttered when an oscillation is transmitted from the oscillation transmitter to the vane .

Brief Summary Text - BSTX (26):

Preferably, the oscillating vane comprises a vane plate, a fixing member for the vane plate and a second vibrational stress dispersing means interposed between the vane plate and the fixing member.

Brief Summary Text - BSTX (27):

Preferably, the oscillating vane is attached to a vane attaching portion of the oscillation transmitter, the vane attaching portion extends in one

United States Patent [19]

Omasa

[11] Patent Number: 5,730,856

[45] Date of Patent: Mar. 24, 1998

[54] METHOD FOR TREATING WASTE LIQUID WITH ELECTROLYTIC OXIDATION AND APPARATUS FOR CARRYING OUT THE SAME

[75] Inventor: Ryushin Omasa, Fujisawa, Japan

[73] Assignee: Nihon Techno Kabushiki Kaisha, Tokyo, Japan

[21] Appl. No.: 560,321

[22] Filed: Nov. 17, 1995

[30] Foreign Application Priority Data

Jul. 25, 1995 [JP] Japan 7-209189

[51] Int. Cl.[6] C02F 1/461

[52] U.S. Cl. 205/742; 205/771; 205/548; 204/222; 204/242; 204/273

[58] Field of Search 205/742, 548, 205/771; 204/222, 242, 273

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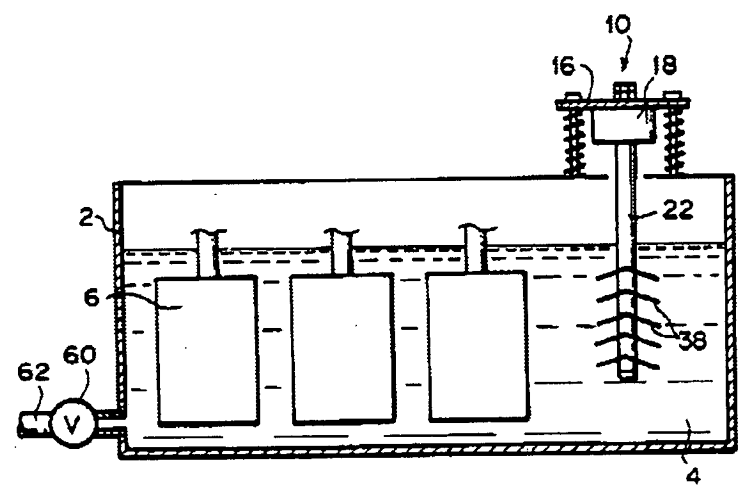
Primary Examiner—Arun S. Phasge
Attorney, Agent, or Firm—Kane,Dalsimer,Sullivan,Kurucz,Levy,Eisele and Richard, LLP

[57] ABSTRACT

An electroless nickel plating waste liquid (4) accommodated in an electrolytic oxidation tank (2) is treated by electrolytic oxidation with vibrating and fluidizing the waste liquid by means of an oscillating stirrer (10) to recover nickel by the electrolytic oxidation as well as recover the waste liquid in which an active component for fertilizer including phosphorus remains as a fertilizer solution. When the waste liquid is neutralized by a neutralizer including an active component for fertilizer such as potassium, nitrogen, etc., the fertilizer solution having higher content of the active component is obtained. The oscillating stirrer comprises an oscillation generator including a 25 to 500 Hz oscillating motor (18), an oscillating rod (22) connected to the oscillation generator, and multistage oscillating vanes (38) attached to the oscillating rod, and the oscillating vane is such that a tip end portion of the vane is fluttered when an oscillation is transmitted from the oscillating rod to the vane.

18 Claims, 12 Drawing Sheets



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EAST - [Default EAST Workspace (Flat Panel LANDSCAPE) wsp 1]

File View Edit Tools Window Help

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  - L1: (268) (205/148).CCLS.
  - L2: (48961) vane or vanes
  - L3: (6) 11 and 12
  - L4: (70421) vibrate or vibrates or vibrated or vibrati
  - L5: (184265) vibration or vibrations or vibrational
  - L6: (211397) 14 or 15
  - L7: (22) 11 and 16
  - L8: (122) (205/104).CCLS.
  - L9: (0) 18 and 12
  - L10: (11) 18 and 16
  - L11: (424) (204/273).CCLS.
  - L12: (31937) electroplat$ or electrodeposit$
  - L13: (3397) (electrolytical$ or electrochemical$) near
  - L14: (33696) 112 or 113
  - L15: (125) 111 and 114
  - L16: (16) 115 and 12
  - L17: (29) 115 and 16
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EAST Browser - L18: (19) 17 not (3 or ...) | US 4229276 A | Tag: S | Doc: 16/19 (SORTED) | Format : KWIC

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11	US 4954235 A	17	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
12	US 4441965 A	9	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
13	US 4425204 A	5	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
14	US 4329209 A	16	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
15	US 4316786 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
16	US 4229276 A	8	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
17	US 4172780 A	10	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT

US-PAT-NO: 4229276

DOCUMENT-IDENTIFIER: US 4229276 A

TITLE: Vibratory plating apparatus

---------- KWIC ---------

Abstract Text - ABTX (1):

A vibratory plating apparatus having a plating liquid in a plating container, a base on which the container is mounted for vibrating movement, and a lid for the top of the container. The bottom of the container has an arcuate bottom. A vibration motor vibrates the container and electrodes are disposed within the container. With this plating apparatus, workpieces can be plated uniformly in a very short period of time at a high current density and with a small amount of plating liquid. Moreover, it eliminates troublesome steps of manually suspending and removing workpieces from hangers.

Brief Summary Text - BSTX (3):

The present invention relates to a vibratory plating apparatus which improves plating efficiency by vibrating a plating container to maintain a uniform concentration of metal ions in the vicinity of cathodes.

Brief Summary Text - BSTX (6):

In a conventional plating apparatus using a barrel, workpieces are installed in a plating liquid in the barrel and the barrel is rotated or rocked to bring the workpieces into sliding contact with cathodes to subject the workpieces to electrodeposition . Barrel type plating apparatuses include one using an open-top bottle type barrel which is rotated while being inclined, and another one using a rectangular or cylindrical barrel having small apertures in the entire wall thereof, horizontally disposed in a plating liquid, and rotated or rocked with the plating liquid to effect the plating of the workpieces. The former is called an inclined barrel type plating apparatus and the latter a horizontal barrel type plating apparatus. In the inclined barrel type plating apparatus, the temperature and the concentration of the plating baths are frequently subject to changes, the insertion and withdrawal of workpieces cannot be easily carried out and anodes having a large surface area cannot be employed.

Brief Summary Text - BSTX (8):

Plating with 3-4.mu. of nickel cannot be carried out efficiently in the horizontal barrel type plating apparatus or it usually requires 60-90 minutes of electrodeposition even if the diameter of apertures in the side wall of the plating barrel are enlarged. In addition, the amount of plating liquid removed from the plating barrel in this apparatus is larger than that in a widely used plating method carried out with workpieces suspended in a plating liquid. This causes the composition of the plating bath to be changed. In other words, quality control cannot be carried out easily. This large amount of plating liquid removed from the plating barrel has caused troubles in the treatment of

United States Patent [19]

Kobayashi et al.

[11] 4,229,276

[45] Oct. 21, 1980

[54] VIBRATORY PLATING APPARATUS

[75] Inventors: Hisamine Kobayashi, Nagoya; Motoo Kawasaki, Hirakata; Shozo Mizumoto, Takarazuka; Hidemi Nawafune, Takatsuki; Sadamasa Suzuki, Tatsuta, all of Japan

[73] Assignee: Shikishima Tipton Manufacturing Co. Ltd., Nagoya; Motoo Kawasaki, Hirakata, both of Japan

[21] Appl. No.: 16,144

[22] Filed: Feb. 28, 1979

[51] Int. Cl.² C25D 17/02; C25D 17/26

[52] U.S. Cl. 204/222; 204/273

[58] Field of Search 204/222, 223, 213, 273, 204/201

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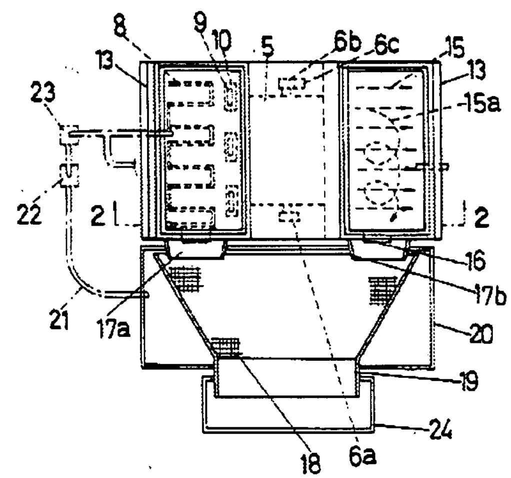
Primary Examiner—T. M. Tufariello

Attorney, Agent, or Firm—Wenderoth, Lind & Ponack

[57] ABSTRACT

A vibratory plating apparatus having a plating liquid in a plating container, a base on which the container is mounted for vibrating movement, and a lid for the top of the container. The bottom of the container has an arcuate bottom. A vibration motor vibrates the container and electrodes are disposed within the container. With this plating apparatus, workpieces can be plated uniformly in a very short period of time at a high current density and with a small amount of plating liquid. Moreover, it eliminates troublesome steps of manually suspending and removing workpieces from hangers.

8 Claims, 8 Drawing Figures



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2	US 6322689 B1	28	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
3	US 6322240 B1	17	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
4	US 6210030 B1	28	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT
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US-PAT-NO: 6322240

DOCUMENT-IDENTIFIER: US 6322240 B1
See image for Certificate of Correction

TITLE: Vibrationally fluidly stirring apparatus

---------- KWIC ---------

Brief Summary Text - BSTX (3):

The present invention relates to a vibrationally fluidly stirring apparatus having a vibration vane which is vibrated in fluid such as liquid to generate vibrational flow in the fluid.

Detailed Description Text - DETX (24):

The vibration vane 10 is preferably formed of thin metal, elastic synthetic resin, rubber or the like, and the thickness thereof may be set so that at least the tip portion of the vane 10 shows a flutter phenomenon (as if it is corrugated) on the basis of the oscillation of the vibration motor 14, whereby the oscillation is applied to the fluid in the tank 13 to cause the vibrational flow . As the material of the metal vibration vane may be used titanium, aluminum, copper, steel, stainless steel, or alloy thereof. As the material of the synthetic resin may be used polycarbonate, vinyl-chloride resin, polypropylene or the like. The thickness is not limited to a specific value, however, in order to transmit the oscillation energy and enhance the effect of the vibration, it is preferably set to 0.2 to 2 mm for metal vibration vane, and 0.5 to 10 mm for plastic or rubber vibration vane. If the thickness is excessively large, the vibrationally fluidly stirring effect is reduced. The vibrational amplitude of the vibration vane 10 is 0.5 to 20 mm for example, preferably 1 to 10 mm.

Detailed Description Text - DETX (26):

Further, all the vibration vanes 10 may be secured perpendicularly to the vibrating bar 7 as shown in FIG. 6A. However, it is preferable that they are secured to be inclined at an angle .alpha. relative to a plane perpendicular to the vibrating bar 7 as shown in FIG. 1. The angle .alpha. is 5 to 30 degrees for example, preferably 10 to 20 degrees in (+) or (-) direction to give the directivity to the vibrational flow of the fluid.

Claims Text - CLTX (12):

a thickness of said vibration vane is se t so that at least a tip portion thereof shows a flutter phenomenon on the basis of an oscilla tion of said vibration motor, whereby said oscillation causes a vibrational flow in said fluid in the tank.

(12) **United States Patent**
Omasa

(10) Patent No.: **US 6,322,240 B1**
(45) Date of Patent: **Nov. 27, 2001**

(54) **VIBRATIONALLY FLUIDLY STIRRING APPARATUS**

(75) Inventor: **Ryushin Omasa**, Fujisawa (JP)

(73) Assignee: **Japan Techo Co., LTD** (JP)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **09/552,194**

(22) Filed: **Apr. 19, 2000**

(30) **Foreign Application Priority Data**

May 7, 1999 (JP) 11-127830

(51) **Int. Cl.**[7] **B01F 11/00**
(52) **U.S. Cl.** **366/118**; 366/256; 366/332; 366/347
(58) **Field of Search** 366/118, 117, 366/243, 255, 256, 257, 258, 259, 260, 276, 347, 332, 333, 334, 335, 242

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European Search Report listing references, EP 00 10 9545.

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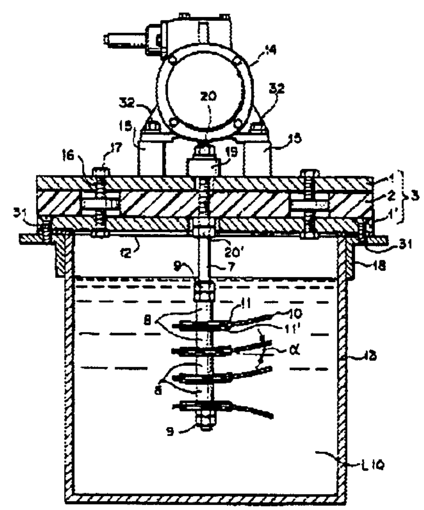
Primary Examiner—Tony G. Soohoo
(74) *Attorney, Agent, or Firm*—Pitney, Hardin, Kipp & Szuch, LLP

(57) **ABSTRACT**

A vibrationally fluidly stirring apparatus includes tank (13) to be charged with liquid (LIQ) to be stirred; a vibration generating portion containing vibration motor (14); vibration absorbing member (3) disposed between the tank (13) and the vibration generating portion; a vibrating bar (7) operationally connected to the vibration generating portion and extended in the tank (13); and a vibration vane (10) attached to the vibrating bar (7), the vibration absorbing member (3) is a laminate of upper metal plate (1), rubber plate (2) and lower metal plate (1').

6 Claims, 10 Drawing Sheets



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64	US 4478689 A	19	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
65	US 4468293 A	11	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
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67	US 4461680 A	10	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
68	US 4436591 A	6	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT

FIG. 1 shows a typical waveform for pulsed deposition;

Detailed Description Text - DETX (15):

Measurements of the chromium content of the alloy at various pH values while also varying the temperature, using the chromium current efficiency as the dependent variable, showed that the efficiency goes through a minimum at about 55.degree. C. and at a pH of 2. At a temperature of 30.degree. C., deposits containing over 18 weight percent chromium are produced by direct current deposition. Significant improvements in deposit morphology are obtained by pulsed electrodeposition, as discussed in greater detail hereinafter.

Detailed Description Text - DETX (16):

Direct current deposition using the plating bath of the invention produces surprisingly and unexpectedly thick nickel-chromium alloy deposits having advantageous morphological characteristics. Either direct or pulsed current deposition according to the process of the invention can give nickel-chromium deposits at least 25 microns thick, and coatings of thicknesses of at least 50 microns, 75 microns, 100 microns, and even 125 microns have been achieved.

Detailed Description Text - DETX (17):

Even better deposits can be obtained using pulsed current deposition. pulsing permits deposition of alloy compositions which cannot be obtained by direct current deposition. Moreover, it is possible to electronically control the coating composition and morphology by control of the pulsed current parameters.

Detailed Description Text - DETX (18):

In pulsed electrodeposition, there are four variables that are of primary importance. These are: (1) pulse height (peak current density); (2) base height (off time current density); (3) on time; and (4) off time.

Detailed Description Text - DETX (20):

It had not been known previously how pulsed current deposition would affect the composition and the morphology of nickel-chromium electrodeposited alloys. The general theory of pulsed current deposition has shown that important affects on morphology and composition of the deposited metal coating can be produced by variation of the operating parameters. During the On Time, the concentration of both nickel and chromium ions is reduced within a certain diffusion distance from the cathode. This so-called diffusion layer pulsates with the same frequency as the applied current. Its magnitude is also related to the peak current density but reaches a limiting value governed primarily by the diffusion coefficient of the ions involved. During the Off Time, the concentration of the reactants builds up again and will reach the equilibrium concentration of the bulk electrolyte if enough time is allowed.

Detailed Description Text - DETX (21):

United States Patent [19]

Lashmore

[11] Patent Number: 4,461,680

[45] Date of Patent: Jul. 24, 1984

[54] PROCESS AND BATH FOR ELECTROPLATING NICKEL-CHROMIUM ALLOYS

[75] Inventor: David S. Lashmore, Frederick, Md.

[73] Assignee: The United States of America as represented by the Secretary of Commerce, Washington, D.C.

[21] Appl. No.: 567,481

[22] Filed: Dec. 30, 1983

[51] Int. Cl.[3] C25D 3/56; C25D 5/14

[52] U.S. Cl. .... 204/41; 204/43 R; 204/43 T; 204/DIG. 9

[58] Field of Search .... 204/43 R, 43 T, 123, 204/DIG. 9, 41

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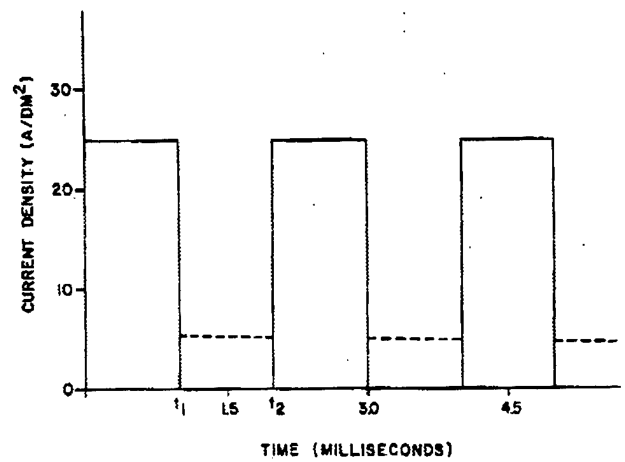
Primary Examiner—G. L. Kaplan
Attorney, Agent, or Firm—Eugene J. Pawlikowski; Alvin J. Englert; Bernhard D. Saxe

[57] ABSTRACT

A process for the electrodeposition of a nickel chromium alloy on a cathodic substrate comprises:
contacting the substrate with an aqueous electrolyte containing: about 50-125 g/l of $CrCl_3.6H_2O$; about 10-125 g/l of $NiCl_2.6H_2O$; about 10-115 g/l of formic acid; about 25-50 g/l of boric acid; and about 50-100 g/l of sodium citrate dihydrate;
adjusting the pH of the bath to about 1-5 and the temperature to about 20°-60° C.; and
passing a sufficient current through the solution and to the substrate to effect deposition thereon of a nickel-chromium alloy.

16 Claims, 4 Drawing Figures



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	Document ID	Pages	1	2	3		5	U	S	C	P	Kind Codes	Sou
62	US 4566953 A	7							☑				USPAT
63	US 4496436 A	9							☑				USPAT
64	US 4478689 A	19							☑				USPAT
65	US 4468293 A	11							☑				USPAT
66	US 4465564 A	6							☑				USPAT
67	US 4461680 A	10							☑				USPAT
68	US 4436591 A	6						☑					USPAT

FIG. 1 shows a typical waveform for pulsed deposition;

Detailed Description Text - DETX (15):

Measurements of the chromium content of the alloy at various pH values while also varying the temperature, using the chromium current efficiency as the dependent variable, showed that the efficiency goes through a minimum at about 55.degree. C. and at a pH of 2. At a temperature of 30.degree. C., deposits containing over 18 weight percent chromium are produced by direct current deposition. Significant improvements in deposit morphology are obtained by pulsed electrodeposition, as discussed in greater detail hereinafter.

Detailed Description Text - DETX (16):

Direct current deposition using the plating bath of the invention produces surprisingly and unexpectedly thick nickel-chromium alloy deposits having advantageous morphological characteristics. Either direct or pulsed current deposition according to the process of the invention can give nickel-chromium deposits at least 25 microns thick, and coatings of thicknesses of at least 50 microns, 75 microns, 100 microns, and even 125 microns have been achieved.

Detailed Description Text - DETX (17):

Even better deposits can be obtained using pulsed current deposition. Pulsing permits deposition of alloy compositions which cannot be obtained by direct current deposition. Moreover, it is possible to electronically control the coating composition and morphology by control of the pulsed current parameters.

Detailed Description Text - DETX (18):

In pulsed electrodeposition, there are four variables that are of primary importance. These are: (1) pulse height (peak current density); (2) base height (off time current density); (3) on time; and (4) off time.

Detailed Description Text - DETX (20):

It had not been known previously how pulsed current deposition would affect the composition and the morphology of nickel-chromium electrodeposited alloys. The general theory of pulsed current deposition has shown that important affects on morphology and composition of the deposited metal coating can be produced by variation of the operating parameters. During the On Time, the concentration of both nickel and chromium ions is reduced within a certain diffusion distance from the cathode. This so-called diffusion layer pulsates with the same frequency as the applied current. Its magnitude is also related to the peak current density but reaches a limiting value governed primarily by the diffusion coefficient of the ions involved. During the Off Time, the concentration of the reactants builds up again and will reach the equilibrium concentration of the bulk electrolyte if enough time is allowed.

Detailed Description Text - DETX (21):

ings exceeded the wear performance of electrodeposited nickel.

The current density, the Duty Cycle and the Period each have an affect on the deposit. Variation of current density for various Duty Cycles, using a fixed On Time of 2 milliseconds, shows that chromium content increases as current density increases, for all Duty Cycles from 20 to 90% (FIG. 2). The higher the Duty Cycle, the higher the chromium content, for the same current density. Variation of the period at a fixed Duty Cycle of 20% and at various current densities resulted in variation of the chromium content (FIG. 3). Local maxima can be seen for periods in the millisecond range.

The base height can be positive, negative or zero, the latter being convenient for studying variations in other parameters. Negative base current density provides cathodic protection by reducing corrosion processes during Off Time. Positive base current density can be useful to reduce surface roughening, especially when plating near the limiting current density of the system.

There is a significant difference in the surface morphology of alloy deposits produced with pulsed galvanostatic deposition, as a function of different waveforms. In general, direct current deposit will produce a rougher surface than pulsed current deposition for the same current density. The Duty Cycle also affects the surface roughness, and it is generally the case that smoother deposits are produced at lower duty cycles. Also, shorter periods generally produce smoother coatings for the same Duty Cycle. Control of pH and temperature to keep these factors substantially constant will also produce a more uniform deposit.

For most pulsed electrodeposition applications it will be advantageous to set the relevant parameters in the following ranges:

Parameter	Broad	Preferred
Period (msec)	0.25-10	0.1
Duty Cycle (%)	20-80	25-50
Current Density (A/dm²)	5-100	25-50
Base Height (A/dm²)	−10–+10	0

It will be appreciated that the foregoing ranges are illustrative and not limitative of the broad scope of this invention. A wide range of variability is possible for the pulsed deposition process, permitting extensive control of the process and allowing the chromium content, the surface morphology and the layering of the deposit to be controlled. This in turn translates into significant control over the resultant properties of the alloy coating. Depending upon the ultimate use, the properties of greatest concern can be controlled to maximize the desired characteristics.

The electrodeposition process of the invention can be effected using standard cells and electrodes, e.g., rotating disc electrodes. Preferably, anodic reactions are minimized by the use of anode/cathode surface area ratios of at least 2/1, and by the use of low polarization anodes. A nickel, nickel-chromium or platinum anode can be used, as can the usual commercial anodes, e.g., lead, graphite, platinized titanium and the like. Anodic reactions are generally not important but separate anode and cathode compartments can be used to prevent diffusion of anodic decomposition products into the cathode plating compartment. For example, semipermeable membranes, e.g., Nafion membranes, can be used to separate the anodic and cathodic regions, espe-

cially when readily decomposable complexing agents, e.g., glycine, are used.

Without further elaboration, it is believed that one skilled in the art can, using the preceding description, utilize the present invention to its fullest extent. The following preferred specific embodiments are, therefore, to be construed as merely illustrative, and not limitative of the remainder of the disclosure in any way whatsoever. In the following examples, all temperatures are set forth uncorrected in degrees Celsius; unless otherwise indicated, all parts and percentages are by weight.

EXAMPLE 1

Comparative Test of Throwing Power

The throwing powers of a plating bath according to the present invention and a nickel-chromium sulfate plating bath according to U.S. Pat. No. 3,888,744 (Stromatt) were compared under identical conditions in a standard Hull Cell, at 3 amps DC for 5 minutes. Each bath was prepared using standard shelf reagents and distilled water. The Stromatt bath had the composition shown in Example I of the patent, 0.15 M NaEDTA, pH 2.4. The bath according to the present invention had pH 2, and the composition:

Constituent	Amount
$NiCl_2.6H_2O$	30 g/l
$CrCl_3.6H_2O$	100 g/l
Formic Acid (95%)	40 ml/l
NaBr	15 g/l
NH_4Cl	50 g/l
Boric Acid	30 g/l
Glycolic Acid	50 g/l
Sodium Citrate Dihydrate	50 g/l
Triton X-100 (100%)	1 drop/l

The Stromatt bath plated out to a distance of 49 mm, while the bath according to the invention plated out to a distance of 71 mm with improved surface morphology, showing the significantly greater throwing power of the present bath.

EXAMPLE 2

Microhardness

Microhardness measurements were carried out with a Vickers indenter at the indicated load of 25 or 50 grams, using a calibrated standard periodically to insure proper operation of the instrument.

$NiCl_2.6H_2O$	30 g/l
$CrCl_3.6H_2O$	100 g/l
NaBr	15 g/l
$Na_3C_6H_5O_7.2H_2O$ (Sodium Citrate Dihydrate)	50 g/l
H_3BO_3	40 g/l
HCOOH (95%)	30 ml/l

Electrodeposition was effected at about 35° C. and at pH 3.5, with zero Amp Off Time current, in a Pyrex cell containing about 0.5 liter of electrolyte, using either platinum or high-purity carbon anodes. The samples were coated rotating disc electrodes about 1 cm in diameter. Other diameters, as well as other geometries, have also been used successfully.

The results are shown in Table I:

5/2003 SN 09/864,650 line 5

EAST Browser - L7: (34) 1 not 5 | US 3480522 A | Tag: S | Doc: 16/34 (SORTED) | Format : FULL

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11	US 3909404 A	3						✓				USPAT
12	US 3804725 A	8						✓				USOCR
13	US 3692639 A	5						✓				USOCR
14	US 3634212 A	7						✓				USPAT
15	US 3503860 A	4						✓				USOCR
	US 3480522 A	6						✓				USOCR
17	US 3450605 A	4					✓					USPAT

US-PAT-NO: 3480522

DOCUMENT-IDENTIFIER: US 3480522 A

TITLE: METHOD OF MAKING MAGNETIC THIN FILM DEVICE

DATE-ISSUED: November 25, 1969

INVENTOR: BROWNLOW JAMES M

US-CL-CURRENT: 205/104, 204/273 , 205/148 , 205/176 , 205/239 , 205/90 , 205/922 , 428/635 , 428/680 , 428/928

Nov. 25, 1969 J. M. BROWNLOW 3,480,522 METHOD OF MAKING MAGNETIC THIN FILM DEVICE Filed Aug. 18, 1066 2- Sheets-Sheet 1 FIG. I ,-i2 34 20 26 22A 30 28 20 10 18 16 18 INVENTOR JAMCLS M. BROWNLOW By CY ATTORNEY

Nov. 25, 1969 J. M. BROWN LOW 3,480,522 METHOD OF MAKING MAGNETIC THIN FILM DEVICE Filed Aug. 18, 1966 2 Sheets-Sheet 2 FIG. 2 34 216 24 22A -30 3 2 2 8 14 20 18 16 42 40 FIG. 4 42 40 i4 ON- PLATING OFF ON-, AGITATION OFF i 0 10 20 30 40 50 60 FIG. 3 TIME/SEC'S

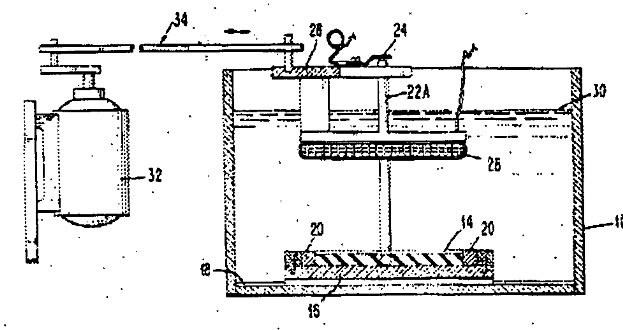
Un'ited States Patent Office' 304800522 3,480,522 METHOD OF MAKING MAGNETIC THIN FILM DEVICE James M. Brownlow, Crompond, N.Y., assignor to International Business Machines Corporation, Armonk, N.Y., a corporation of New York Filed Aug. 18,1966, Ser. No. 573,417 Int. Cl. C23b 5150; C22c 39112 U.S. Cl. 204-40 18 Claims ABSTRACT OF THE DISCLOSURE The magne'lic nickel iron film element is plated from a- relatively dilute aqueous bath. A pulse plating technique is employed with a series of current pulses being applied to the bath and the bath being agitated only during the time between current pulses The bath includes, in addition to the nickel and iron ions, copper ions. Each current pulse plates two layers. The first layer is a nickel iron alloy which is rich in copper and is non- magnetic and the second layer is a nickel iron alloy which has a low percenta.-e of copper and is magnetic. The magnetic storage element includes a plurality of such alternate magnetic and non ma.-netic layers. The present inven@ion relates to magnetic film devices and more partictilarly to improved magnetic alloy thin film structures as well as methods of fabricating alloy ma.- netic thin film structures. Though magnetic film structures have many useful applications outside the computer field, a principal commercial use of 'these structures is in lar.-e scale digital computers and within computers the primary use of devices fabricated using magnetic films is in large scale memories. Further thou.-h both thick and thin film type devices have been developed for computer memory applications, by far the most significant use, both present and contemplated, is in anisotropic thin magnetic film elements. By the term anisotropic it is meant that the films are so prepared that the magnetic moments in the film, in the absence of an applied tield, align themselves parallel to a direction in the film usually termed the easy axis Magnetic films of this type which are termed thin films usually have a thickness less than 10,000 angstroms, and can have the magnetic moments switched from one orientation to another by high speed rotation during which process the film behaves essentially as a single magnetic domain. The present invention, both as to structure and method, is not limited ot anisoptric thin films used in memory applications, but since the principal use of magnetic films is in this area, this is the principal application of the invention as

Nov. 25, 1969 J. M. BROWNLOW 3,480,522

METHOD OF MAKING MAGNETIC THIN FILM DEVICE

Filed Aug. 18, 1966 2 Sheets-Sheet 2

FIG. 2



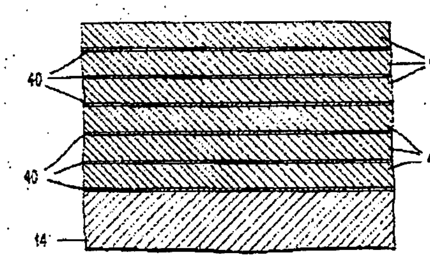


FIG. 4

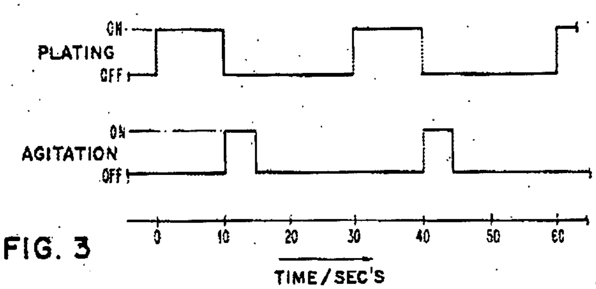


FIG. 3

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	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor
1	US 6547944 B2	20030415	9	Commercial plating of nanolaminates	205/96	205/102; 205/103;		Schreiber, Chris M. et al.
2	US 6312579 B1	20011106	5	Bearing having multilayer overlay and method of	205/95	205/102; 205/104;		Bank, Brian L. et al.
3	US 5730852 A	19980324	8	Preparation of cuxinygazsen (X=0-2, Y=0-2, Z=0-2, N=0-3)	205/192	136/264; 136/265;		Bhattacharya, Raghu N. et al.
4	US 5156729 A	19921020	6	Method of making a plain bearing sliding layer	205/104	205/149; 205/176;		Mahrus, Duraid et al.
5	US 5132200 A	19920721	6	Electrophotographic photoreceptor with porous	430/131	205/104; 205/201;		Fukuda, Yuzuru et al.
6	US 4849302 A	19890718	8	Electrolytically metallized article and processes	428/621	205/104; 205/118;		Ostwald, Robert
7	US 4049507 A	19770920	4	Electrodepositing method	205/104	204/273; 204/DIG.9;		Tokumoto, Shin-Ichi et al.
8	US 4046643 A	19770906	21	Production of multi-metal particles for powder	205/103	204/223; 205/104;		Rippere, Ralph E.
9	US 4032413 A	19770628	4	Electroplating bath and method for the	205/103	205/104; 205/148;		Dotzer, Richard et al.
10	US 4000046 A	19761228	9	Method of electroplating a conductive layer over an	205/103	205/102; 205/104;		Weaver, Charles A.
11	US 3909404 A	19750930	3	Composition and process for electrodepositing a black	205/104	205/243; 205/285		Boycott, William A.
12	US 3804725 A	19740416	8	METHODS AND APPARATUS FOR TREATING AN ARTICLE	205/96	204/229.5; 204/DIG.9;		

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  - L17: (209643) 111 or 114
  - L20: (706535) current or voltage
  - L23: (24409) 117 near3 120
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	U	1	PT	P	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor	B	C	2	3		5
1	☑	☐	☐	☐	JP 10092602 A	19980410	7	METALLIC FILM RESISTOR AND ITS MANUFACTURE				MIKAMOTO, NAOHIRO et al.	☐	☐	☐	☐	☐	☐
2	☑	☐	☐	☐	JP 09059797 A	19970304		AUTOMATIC LIQUID TREATING LINE				OBA, KAZUO et al.	☐	☐	☐	☐	☐	☐
3	☑	☐	☐	☐	JP 62235714 A	19871015		FORMATION OF MAGNETIC ALLOY THIN FILM AND DEVICE				YOSHIDA, TOSHIHIRO et al.	☐	☐	☐	☐	☐	☐

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2	JP 10092602 A	7						☑					JPO
3	JP 09059797 A							☑					JPO

PAT-NO: JP362235714A

DOCUMENT-IDENTIFIER: JP 62235714 A

TITLE: FORMATION OF MAGNETIC ALLOY THIN FILM AND DEVICE THEREFOR

PUBN-DATE: October 15, 1987

INVENTOR-INFORMATION:
NAME
YOSHIDA, TOSHIHIRO
KOBAYASHI, TETSUO

ASSIGNEE-INFORMATION:
NAME COUNTRY
HITACHI LTD N/A

APPL-NO: JP61078204

APPL-DATE: April 7, 1986

INT-CL (IPC): H01F041/26, C25D005/08

ABSTRACT:

PURPOSE: To form a magnetic alloy thin film, having uniform compositional distribution, on a stepped substrate by a method wherein the circulation of a plating solution, the agitation of the plating solution in the vicinity of the substrate surface, and a pulse superposition DC power source are properly combined.

CONSTITUTION: When a magnetic alloy thin film is going to be electroplated on a substrate, a horizontal current circuit, having the crosssectional area corresponding to the shape of the substrate, is formed between an anode plate 24 and a cathode plate 14 droopingly provided facing each other in a plating cell 25 in which a plating solution is circulated. The plating solution is fed into an inflow chamber 18 from an inflow pipe 17 with a circulating pump 28, it is sent to a cathode chamber 12 passing through an inflow hole 19, the plating solution in the cathode chamber 12 is agitated by an agitating spatula 20, and sent to an anode chamber 10 passing through an aperture part 15. A pulse superposition DC plating power source 36, with which a current wherein a rectangular pulse is DC-superposed is supplied through the intermediary of a lead, is connected to the point located between the anode plate 24 and the cathode plate 14.

⑲日本国特許庁(JP)　⑪特許出願公開

⑫公開特許公報(A)　昭62-235714

⑤Int.Cl.⁴	識別記号	庁内整理番号	⑬公開 昭和62年(1987)10月15日
H 01 F 41/26		7354-5E	
C 25 D 5/08		7325-4K	

審査請求 未請求 発明の数 2 (全5頁)

㊿発明の名称　磁性合金薄膜の形成方法及びその装置

㉑特　願　昭61-78204
㉒出　願　昭61(1986)4月7日

㉒発明者　吉田　敏博　小田原市国府津2880番地　株式会社日立製作所小田原工場内
㉒発明者　小林　哲夫　小田原市国府津2880番地　株式会社日立製作所小田原工場内
㉑出願人　株式会社日立製作所　東京都千代田区神田駿河台4丁目6番地
㉔代理人　弁理士　秋本　正実

明　細　書

1．発明の名称

磁性合金薄膜の形成方法及びその装置

2．特許請求の範囲

1．磁性合金薄膜を基板上に電気めっきするめっきセル内にめっき液を循環させ、該めっきセル内に対向配設した陽極板と陰極板間に該陰極板にセットした基板の形状に応じた断面積を有する水平電流路を集束形成せしめるとともに、該陰極板付近でめっき液を撹拌し、かつ陽極板と陰極板間にパルス電流を直流電流に重畳させた電流を供給して電気メッキにより磁性合金薄膜を基板上に形成する磁性合金薄膜の形成方法。

2．磁性合金薄膜を基板上に電気めっきするめっきセルと、該めっきセル内にめっき液を循環させる手段と、上記めっきセル内に対向配設した陽極板と陰極板の該陰極板に基板をセットする手段と、上記陽極板と陰極板間に基板の形状に応じた断面積を有する水平電流路を集束形成せしめる手段と、上記陰極板付近でめっき液を撹拌する手段と、上記陽極板と陰極板間にパルス電流を直流電流に重畳させた電流を供給して電気めっきにより磁性合金薄膜を基板上に形成するめっき電源とを備えた磁性合金薄膜の形成装置。

3．発明の詳細な説明

〔産業上の利用分野〕

本発明は磁性合金薄膜の形成方法及びその装置に係り、特に誘導型薄膜磁気ヘッド用等の磁性を付与した段差のある基板上に均一な合金組成を有する磁性合金薄膜を再現性よく容易にめっきすることのできる磁性合金薄膜の形成方法及びその装置に関する。

〔従来の技術〕

従来より磁性合金薄膜とくに約81Ni−19Feの合金組成を有するパーマロイ薄膜は、低い保磁力と高周波領域での高い透磁率を示すので薄膜磁気ヘッドなどの高速スイッチング素子用磁性材料として広く使用されている。たとえば第8図に示すような断面の構造を有する誘導型薄膜磁気ヘッド

5/2003 SN 09/864,650 EPO, JPO line 53

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3	JP 09059797 A	8		JPO

PAT-NO: JP409059797A

DOCUMENT-IDENTIFIER: JP 09059797 A

TITLE: AUTOMATIC LIQUID TREATING LINE

PUBN-DATE: March 4, 1997

INVENTOR-INFORMATION:
NAME
OBA, KAZUO
SHIMA, YOSHINORI
OBA, AKIRA

ASSIGNEE-INFORMATION:

NAME	COUNTRY
SAKAE DENSHI KOGYO KK	N/A

APPL-NO: JP07217757

APPL-DATE: August 25, 1995

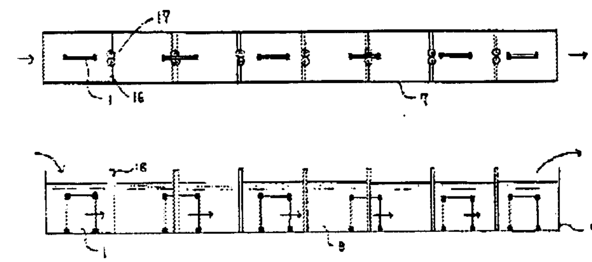
INT-CL (IPC): C25D021/12, C25D019/00

ABSTRACT:

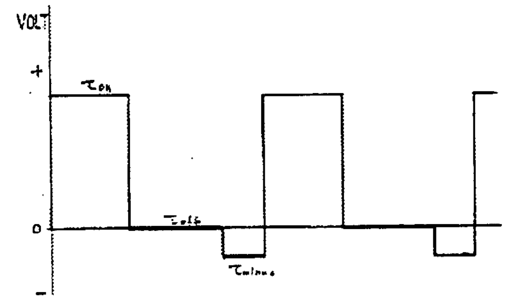
PROBLEM TO BE SOLVED: To efficiently and automatically surface-treat a plateshaped material by detecting the size of the material continuously conveyed in a horizontal position, setting the material in a vertical position and introducing the material into a liq. treating tank corresponding to the size.

SOLUTION: A plate-shaped material 1 such as a printed circuit board is cleaned, bored and so on as pretreatment, the size is detected, the four corners are held by jigs 5 and 6, and then the material is set in a vertical position, introduced into a plating tank corresponding to the size and vertically traveled on a rail 15. Air is blown from a bottom pipe 11 to agitate a plating soln. 8, and a pulse current is applied with the material 1 as a cathode to electroplate the surface. A metallic-mesh cylinder packed with many metallic balls of copper, etc., is used as an anode 9 and made vertically slidable in accordance with the size of the material 1 and the amt. of the soln. 8. Consequently, a current is applied on the entire surface of the material 1 at a uniform density, and a plating film uniform in thickness is formed on the entire surface of the material 1.

【図6】



【図7】



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11	US 5188718 A	7	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
12	US 5186803 A	5	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT
13	US 5167779 A	16	☐	☐	☐	☐	☐	☐	☑	☐	☐		USPAT

US-PAT-NO: 5167779

DOCUMENT-IDENTIFIER: US 5167779 A

TITLE: Process and apparatus for electrolyte exchange

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Current US Cross Reference Classification - CCXR (1):

204/201

US005167779A

United States Patent [19]

Henig

[11] Patent Number: 5,167,779

[45] Date of Patent: Dec. 1, 1992

[54] PROCESS AND APPARATUS FOR ELECTROLYTE EXCHANGE

[76] Inventor: Hans J. Hasig, Albrecht Achilles Str. 48, Nürnberg, Fed. Rep. of Germany, 8500

[21] Appl. No.: 569,124

[22] Filed: Aug. 17, 1990

[51] Int. Cl.[5] C25D 5/00

[52] U.S. Cl. 204/140; 205/149; 205/150; 204/201; 204/222; 204/214

[58] Field of Search 204/222, 285, 201, 214, 204/287, 140, 24, 27; 205/149, 150

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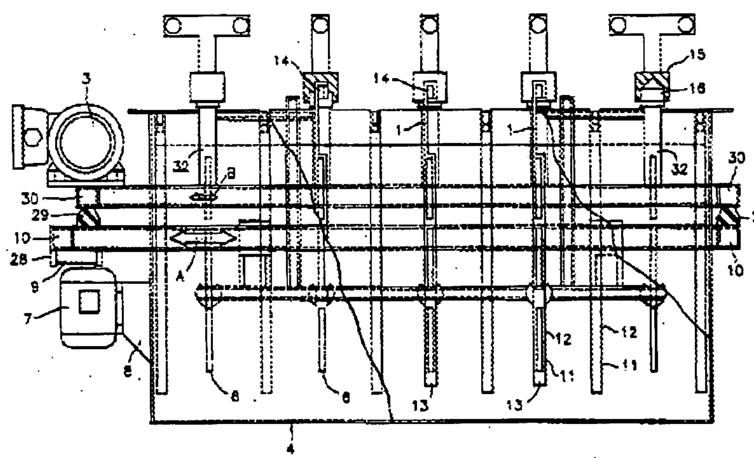
Primary Examiner—John Niebling
Assistant Examiner—Arun S. Phasge
Attorney, Agent, or Firm—Flelt, Jacobson, Cohn, Price, Holman & Stern

[57] ABSTRACT

A process and apparatus for obtaining an intensive and continuous exchange of electrolyte on the surfaces of workpieces (11, 21) subjected to chemical or electrolytical treatments wherein the exchange is achieved by subjecting the workpieces (11, 21) to strong pulsating oscillations of a frequency of at least 1 Hz and of an amplitude of less than 10 mm, when immersed in the electrolyte. The vibrations are transmitted from an oscillation generator (3) mounted on a workpiece carrier, such as a rack (1, 4) or a rotating dipping drum 92). These high frequency low amplitude oscillations may be combined with low frequency, relatively long oscillations (7, 9, 10, 18), occurring simultaneously. The oscillation generator (3) is arranged on the individual transportable workpiece carriers (1, 2, 14). The workpieces may be boards (11) for printed circuits having through holes (12) or a pourable mass of parts (21).

28 Claims, 8 Drawing Sheets



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US-PAT-NO: 5755948

DOCUMENT-IDENTIFIER: US 5755948 A

TITLE: Electroplating system and process

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Current US Cross Reference Classification - CCXR (1):

204/201

US005755948A

United States Patent [19]

Lazaro et al.

[11] Patent Number: 5,755,948

[45] Date of Patent: May 26, 1998

[54] ELECTROPLATING SYSTEM AND PROCESS

[75] Inventors: Anton Ernest Lazaro, Park Ridge; Peter Heinrich Tremmel, Chicago, both of Ill.

[73] Assignee: Hardwood Line Manufacturing Co., Chicago, Ill.

[21] Appl. No.: 786,872

[22] Filed: Jan. 23, 1997

[51] Int. Cl.^{6} C25D 5/00; C25D 7/00; C25D 17/00; C25B 15/00

[52] U.S. Cl. 205/143; 205/145; 204/201; 204/213; 204/237

[58] Field of Search 205/143, 145; 204/201, 213, 237

[56] References Cited

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Primary Examiner—Kathryn L. Gorgos

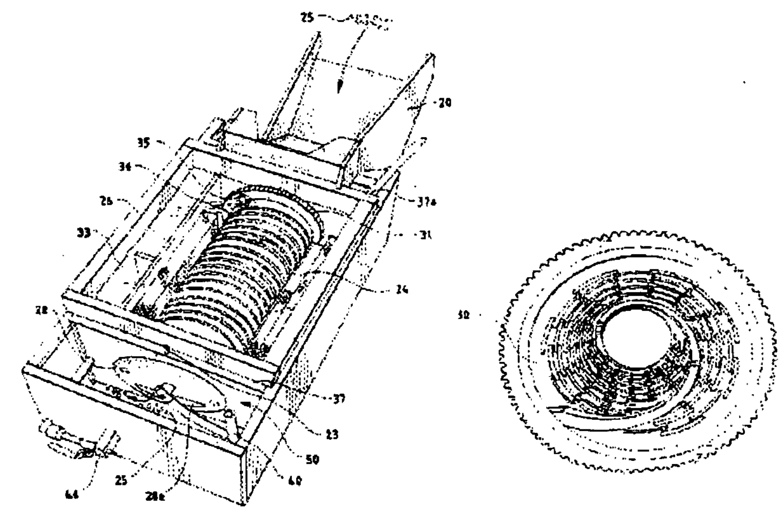
Assistant Examiner—Edna Wong

Attorney, Agent, or Firm—Irwin C. Alter

[57] ABSTRACT

This invention relates to a system and process for electroplating that has plating drums whereby the plating drums are filled, and emptied of parts at each individual plating cell and the parts are cleansed by spraying, wiping and/or drying and whereby only cleansed parts are transported from plating cell to plating cell. This results in less contamination and higher efficiencies in plating.

15 Claims, 9 Drawing Sheets



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US-PAT-NO: 6551483

DOCUMENT-IDENTIFIER: US 6551483 B1

TITLE: Method for potential controlled electroplating of fine patterns on semiconductor wafers

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Abstract Text - ABTX (1):

Controlled-potential electroplating provides an effective method of electroplating metals onto the surfaces of high aspect ratio recessed features of integrated circuit devices. Methods are provided to mitigate corrosion of a metal seed layer on recessed features due to contact of the seed layer with an electrolyte solution. The potential can also be controlled to provide conformal plating over the seed layer and bottom-up filling of the recessed features. For each of these processes, a constant cathodic voltage, pulsed cathodic voltage, or ramped cathodic voltage can be used. An apparatus for controlled-potential electroplating includes a reference electrode placed near the surface to be plated and at least one cathode sense lead to measure the potential at points on the circumference of the integrated circuit structure.

Brief Summary Text - BSTX (14):

Controlling the potential during electroplating provides a process to mitigate corrosion of a metal seed layer on recessed features in a layer of an integrated circuit structure. The corrosion is due to contact of the seed layer with an electrolyte solution during the entry phase. The potential can also be controlled to provide conformal plating over the seed layer during the initiation phase and bottom-up filling of the recessed features during the third, bottom-up filling phase. At each phase, a constant cathodic voltage, pulsed cathodic voltage, or ramped cathodic voltage can be used.

Detailed Description Text - DETX (28):

EXAMPLE B: Application of a pulsed cathodic voltage having a value from about -10 mV to about -500 mV with respect to a reference electrode, having a waveform period from about 0.1 ms (milliseconds) to about 10 ms and a duty cycle from about 1% to about 50%, for a duration of about 0 to about 5 seconds.

Detailed Description Text - DETX (35):

EXAMPLE D: Apply a pulsed cathodic voltage of from approximately -10 mV to approximately -1000 mV having a waveform period of about 0.1 to 10 ms and a duty cycle of approximately 1% to 50%. A background cathodic dc voltage of from about -5 mV to about -100 mV may be optionally superimposed on the pulsed cathodic voltage.

6/18
1541 ||

Detailed Description Text - DETX (37):

We note that the use of a proper shape and length of pulsed potential

US 6,551,483 B1

1

METHOD FOR POTENTIAL CONTROLLED ELECTROPLATING OF FINE PATTERNS ON SEMICONDUCTOR WAFERS

CROSS REFERENCE TO RELATED APPLICATION

This application is a continuation of U.S. patent application Ser. No. 09/515,414, filed on Feb. 29, 2000, and now abandoned.

BACKGROUND OF THE INVENTION

1. Technical Field

This invention relates generally to electroplating methods and apparatus and, in particular, to processes and apparatus for potential-controlled electrodeposition of metal into small integrated circuit features such as vias and trenches.

2. Description of Related Art

Conductive interconnections on integrated circuits typically take the form of trenches and vias. In modern submicron integrated circuits, trenches and vias are typically formed by a "damascene" or "dual damascene" process as described, for example, in the reference *ULSI Technology*, Eds. C. Y. Chang and S. M. Sze (McGraw-Hill, 1996, pp. 444–445.) In damascene processing, an interlayer dielectric (typically SiO_2) is deposited atop a planarized layer containing, for example, a metal via. The top dielectric layer is patterned and etched, typically using conventional photolithographic procedures. Metal is then deposited into features and on the flat field region atop the features, typically first by CVD, PVD and then by electrodeposition. The metal layer is typically planarized resulting in the desired metallic pattern. Dual damascene processing is similar but makes use of two patterning and etching steps and typically fills features with metal spanning more than one layer in a single metallization step. A more complete description of damascene and dual damascene processing is found in the cited reference.

As the art moves towards integrated circuits having reduced feature sizes, it becomes increasingly difficult to form electrically conductive metallizations such as vias, contacts and conductors. Techniques for forming such metallizations include physical vapor deposition (PVD), chemical vapor deposition (CVD), plasma enhanced chemical vapor deposition (PECVD) and electrochemical deposition (also referred to as electroplating or electrodeposition) of metals such as copper. Electroplating is particularly well suited for the formation of small embedded damascene feature metallization due to the ability to readily control the growth of the electroplated film for bottom-up filling without voids, and due to the superior electrical conductivity characteristics of the electroplated film. However, there are also several obstacles which need to be overcome to fully realize these advantages.

One challenge facing damascene and dual damascene processing techniques is the difficulty of initiating the growth of the metal film within recessed features without forming voids or seams. In typical PVD and some CVD processes, metal may preferentially deposit near the top of recessed features leading to a "bottleneck" shape. Further plating of metal onto the bottleneck may result in sealing the top of the feature before completely filling the feature with metal, creating a void. Voids increase the resistance of the conductor over its designed value due to the absence of planned-for conductor. Also, trapped electrolyte in sealed voids may corrode the metal. This may lead to degraded device performance or device failure in extreme cases.

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The problem of electroplating metal into integrated circuit features without the formation of voids or seams is the subject of previously filed U.S. patent application Ser. No. 09/410,170, commonly assigned with the present invention, (hereinafter "prior application"), and now abandoned and incorporated herein by reference. The prior application identified previously unrecognized problems with conventional electroplating techniques and described a four-step approach to overcome them or to mitigate their deleterious effects on the electroplating processes. The four phases relate to the following: 1) the entry of the wafer into the electroplating bath, including procedures to reduce or eliminate corrosion of thin metal seed layers already deposited on the surfaces of recessed features ("entry phase"); 2) the conformal growth of metal seed layers having sufficient thickness to support delivery of current to the bottom of the feature and permit subsequent bottom-up feature filling ("initiation phase"); 3) preferential deposition of metal onto the bottom of high aspect ratio ("AR") features (having AR's greater than about 0.5) leading to bottom-up filling and the reduction of the ARs; and 4) rapid filling of low AR features in a rapid substantially conformal manner until the required metal thickness is achieved.

The methods described in the prior application relate generally to constant current ("galvanostatic") or controlled current ("galvanodynamic") processes. Thus, the current was the parameter controlled and the voltage was understood to take on whatever value necessary to achieve the specified current. Use of galvanostatic or galvanodynamic processes has the advantage of requiring typically simpler and less expensive power sources than those typically required in the practice of controlled-voltage ("potentiodynamic") processes.

Prior art and literature pertaining to potentiostatic and potentiodynamic electroplating is limited in comparison with the literature and prior art related to galvanostatic and galvanodynamic electroplating. Controlled-potential electroplating is typically a more complicated process than controlled current electroplating for several reasons. 1) Controlled-current power supplies are more readily available at lower cost while only a limited number of (typically) more expensive power supplies are available for controlled-potential service. 2) A "four wire" system is typically required in controlled-potential electroplating in order to compensate adequately for potential drop in current-carrying wires and for contact resistances. For applications requiring excellent metal thickness uniformity, such as the integrated circuit fabrication processes described here, a reference electrode probe is advisable.

The present invention describes several advantages to be obtained with the use of controlled-potential electroplating processes for small recessed features in integrated circuit devices. Equipment fully to take advantage of potentiodynamic electroplating processes for the four-step filling of integrated circuit features with metal is also described.

SUMMARY OF THE INVENTION

Controlled-potential electroplating provides an effective method of electroplating metals onto the surfaces of high aspect ratio recessed features of integrated circuit devices. The rate of electroplating is strongly dependent on current density which is correlated with the applied voltage. Thus controlled-potential methods provide tighter control of the electroplating process than controlled-current methods. In addition, controlled-potential electroplating methods are more universally applicable, from wafer to wafer, essentially

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20	US 6129830 A	6	☐	☐	☐	☐	☐	☑	☐	☐	☐		USPAT

Detailed Description Text - DETX (2):

The diagram in FIG. 1a applies to electroplating with direct current. In FIG. 1b the bath current is interrupted for a short time. It remains, however, unipolar i.e. the polarity of the current direction is not reversed. The pulse times lie by preference in the order of magnitude of 0.1 milliseconds up to seconds. The pause times are correspondingly shorter. FIG. 1c shows a unipolar pulse current with different amplitudes. FIG. 1d shows a bipolar current, i.e. a pulse current which is briefly reversed in polarity with a long electroplating time and with a short deplating time. The deplating amplitude here amounts to a multiple of the metallizing amplitude. However, altogether, with an electroplating time of e.g. 10 milliseconds and with a deplating time of 1 millisecond, there is a clear excess of the amount of charge needed for electroplating as opposed to that needed for deplating. This pulse form is particularly suitable for electroplating on both sides printed circuit boards with fine holes. In FIG. 1e, a double pulse form is shown which can be achieved with the method according to the invention. Unipolar pulses here alternate with bipolar pulses .

Detailed Description Text - DETX (3):

The electroplating cell represents for the electroplating current an ohmic load as a good approximation. With a bath current supply according to FIG. 1b, bath current and bath voltage are therefore in phase. The low parasitic inductances of the electrical conductors to the electrolytic cell and back to the current source can be disregarded. Pulse currents contain on the other hand alternating currents. With increasing edge steepness of the pulses, the proportion of the high frequencies of the alternating currents becomes greater. Steep pulse edges have a short pulse rise and fall time. The line inductances represent inductive resistors for these alternating currents. They delay the pulse edges. However these effects are not considered below. They are independent of the type of pulse generation and therefore always the same if special measures are not taken. The simplest measures consist in using electrical lines with very low ohmic and inductive resistances. In the figures, in order to simplify the drawing, the electroplating current is always represented as, or assumed to be, in phase with the voltage.

Detailed Description Text - DETX (4):

FIGS. 2a and 2b show the feeding in, according to the invention, of the compensating pulse current by means of the current transformer 1. The bath direct current source 2 is connected via electrical lines 3 with the electrolytic bath, which is here represented as the bath resistor R.sub.B with the reference number 4. The secondary winding 6 of the current transformer 1 is connected into this high current circuit 5 in series with the electrolytic bath. The primary side 7 of the transformer is fed by the pulse electronic unit 8. The pulse electronic unit 8 is supplied with energy via the main supply 9. The current and voltage paths for the pulses according to FIG. 1d correspond in principle also to the pulse forms of the other diagrams in FIG. 1. They differ only in the momentary size of the compensating current. For this reason the voltages or currents belonging to FIG. 1d are indicated in the following figures and considered.

Detailed Description Text - DETX (6):

U.S. Patent Oct. 17, 2000 Sheet 1 of 7 6,132,584

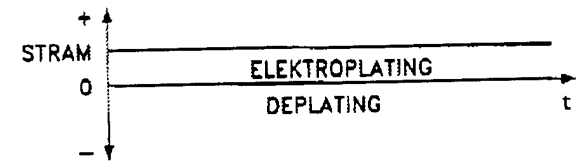

FIG. 1a

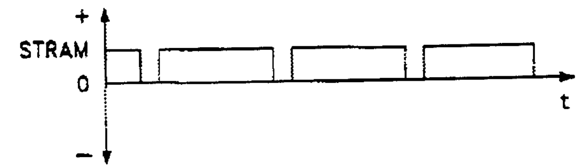

FIG. 1b

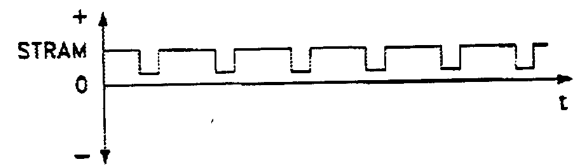

FIG. 1c

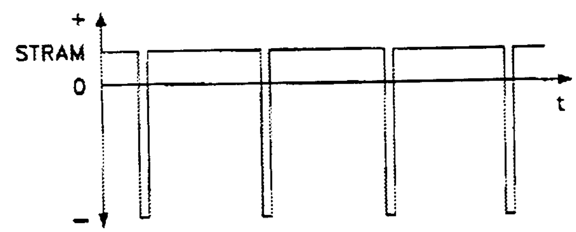

FIG. 1d

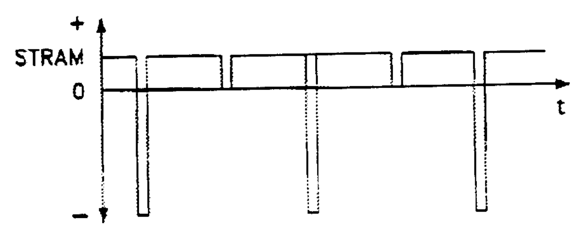

FIG. 1e

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